

## Thermal Expansion Anisotropy of Zr-2.5Nb Pressure Tube Material on Heating to 1100°C

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### Abstract

The anisotropic thermal expansion (and contraction) properties of Zr-2.5Nb pressure tube material have been investigated up to 1100°C using a high-resolution dilatometer. The thermal expansion measured along two principal axes of the pressure tube (longitudinal and transverse directions) are compared with calculated values from a derived expression with material parameters: single-crystal thermal coefficients ( $\alpha$  and  $\beta$  phase), texture, phase volume fractions, and phase transformation strain. The calculated thermal expansion is shown to capture the thermal expansion and anisotropic characteristics throughout the phase transformation regime (two phase region) and into the high-temperature single  $\beta$ -phase domain.

### 1. Introduction

Zr-2.5Nb alloy is used for the pressure tubes in CANDU<sup>®1</sup> reactor fuel channels. The pressure tubes are fabricated by hot extrusion of hollow billets into tubes, which are then cold-drawn (25-30%) and stress relieved for 24 hr at 400°C [1]. The microstructure of the pressure tube consists of elongated hexagonal close-packed (hcp)  $\alpha$ -Zr grains containing a high dislocation density, and a thin layer of body-centered cubic (bcc)  $\beta$ -phase at the  $\alpha$ -grain boundaries. Small quantities of meta-stable  $\omega$  phase may also exist in the material, depending on the thermo-mechanical history. At room temperature, a typical pressure tube contains mostly  $\alpha$ -phase Zr with  $\approx 1$  wt% Nb and a small amount of Nb-stabilized  $\beta$ -phase with  $\approx 20$  wt% Nb [1].

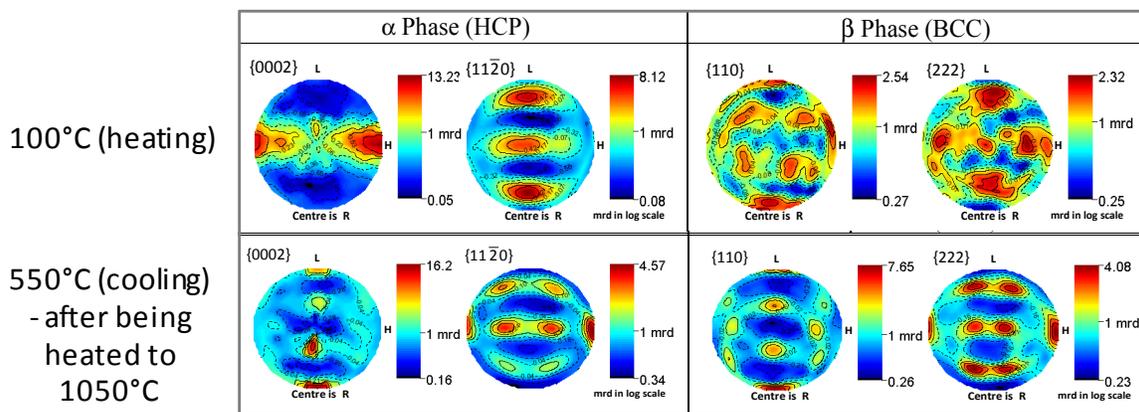
The fabrication procedures used to produce the pressure tubes generate a strong crystallographic texture (Figure 1), and result in anisotropic mechanical properties in the pressure tube materials [1]-[3]. The crystallographic texture of the deformed  $\alpha$ -Zr grains exhibits a large concentration of the basal (0002) plane normals aligned in the hoop (H) (i.e. transverse, T) direction of the tube, and with the prism (11 $\bar{2}$ 0) poles mainly oriented in the tube's radial (R) direction. The tube textures are developed mainly during high-temperature extrusion of the billet [1]. A previous study [4] showed that the pressure tube  $\alpha$ -Zr grain texture is stable on first heating to high temperatures up to 1050°C, and is modified on cooling after being heated to 1050°C (Figure 1). Table 1 gives the Kearns' texture numbers [5] previously determined for the distribution of basal plane normals in the three principal directions of the pressure tube [4]. On heating to high temperatures, the material's microstructure changes as a result of  $\alpha \rightarrow \beta$  phase transformation. On

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cooling back down, a reversal of the phase transformation occurs (i.e.,  $\beta \rightarrow \alpha$ ). Some transformation strains are generally produced during these phase transformation processes [5].

Most metals expand during heating and contract on cooling. If this phenomenon is constrained, large stresses can be generated in the materials with changes in temperature. The calculation of thermal stresses requires this thermo-physical property, which is experimentally determined as the ‘coefficient of thermal expansion’ (CTE). For thermal contraction, CTE values are also generally used for calculating thermal stresses, although the material may not be contracting in the same way as it expands. In a reactor operation, thermal cycling of reactor components such as pressure tubes would occur during start-up (heating) and shut-down (cooling) and thus some thermal stresses would be generated. For fuel elements, thermal stresses can also be generated if the CTE of the fuel and sheath are significantly different.



**Figure 1 Pole figures (equal-area-projection) of hcp  $\alpha$ - and bcc  $\beta$ -phase in as-manufactured Zr-2.5Nb pressure tube at: (a) 100°C on heating, and (b) 550°C on cooling after being heated to 1050°C [4]. (R, L and H are the radial, longitudinal, and hoop (transverse) directions of the tube).**

**Table 1 Kearns (texture) numbers [5] for Zr-2.5Nb pressure tube determined from Figure 1 [4].**  
 (Subscripts R, T and L denote the radial, transverse, and longitudinal directions of the tube)

Zr-2.5Nb pressure tube material	Kearns (texture) numbers		
	$f_R$	$f_T$	$f_L$
As-mfg'd sample at 100°C (heating)	0.26	0.61	0.13
Same as-mfg'd sample at 550°C (cooling) after being heated to 1050°C	0.32	0.18	0.50

In this study, the anisotropic thermal expansion and contraction of Zr-2.5Nb pressure tube material was examined by continuous heating and cooling of samples taken from the tube's longitudinal and transverse direction using dilatometry. The same samples were given two consecutive heating and cooling cycles. This paper summarizes the results of this investigation, showing the predominant effect of texture and phase transformation [4] on the dilatation anisotropy in as-manufactured Zr-2.5Nb pressure tube material during two consecutive thermal cycles.

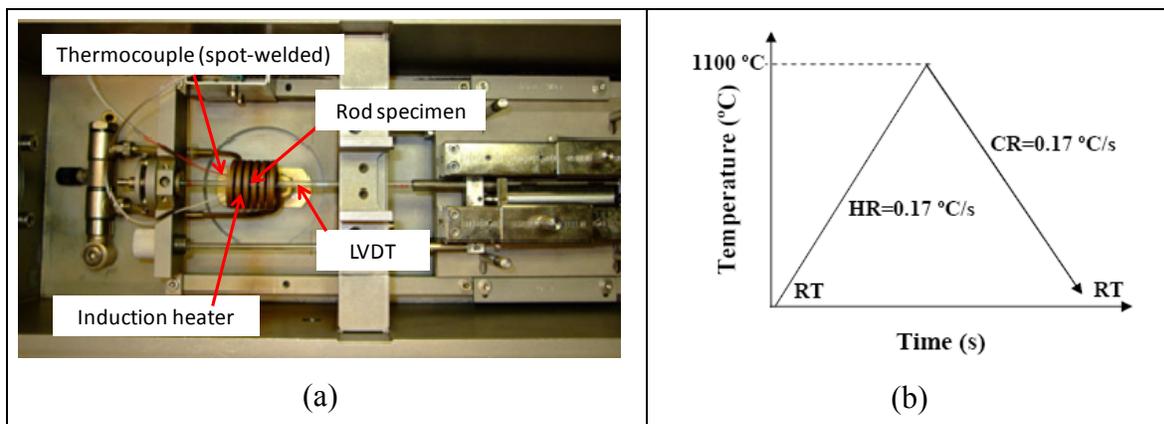
## 2. Experimental

### 2.1 Dilatation Measurements

Solid rod specimens were machined from the transverse and longitudinal directions of the as-manufactured Zr-2.5Nb pressure tube material. The initial dimensions of the transverse and longitudinal rod specimens were 10.017 mm long x 3.786 mm diameter, and 10.078 mm long x 3.781 mm diameter, respectively. A micrometer (MITUTOYO with  $\pm 0.001$  mm resolution) was used to measure the dimensions of the specimens. The change in length of the rod specimen was measured using a high resolution BÄHR Dilatometer (model DIL 805A/D) during heating from room temperature to 1100°C and also on cooling back down to room temperature under inert atmosphere. Figure 2a shows a photograph of the dilatometer equipped with an induction coil heater for heating the sample and an inert gas system for controlled cooling of the sample and the simultaneous measurements of the length and temperature of the sample. The change in length of the rod sample was measured continuously using the LVDT (linear voltage displacement transducer) systems. The LVDT of the dilatometer has a resolution of  $\pm 50$  nm.

In this study, the sample was subjected to a uniform rate of heating and cooling at 0.17°C/s (Figure 2b) with very little tensile or compressive load on the ends of the sample. The dilatations in the same sample of as-manufactured pressure tube material for both longitudinal and transverse directions of the tube were measured using two consecutive heating and cooling cycles (i.e., identified as Run 1 and Run 2). The same rate for heating and cooling was applied in all the test runs.

The length (dilatation) measurements from the LVDT and the temperature readings were sampled at a rate of 30 Hz during the test runs. The data for temperature and length change (i.e., instantaneous length minus the original length) obtained on the test samples are used for analysis to determine: (i) the CTE values and anisotropy, (ii) the fraction and proportion of the  $\alpha$  and  $\beta$  phase material, and (iii) an expression to calculate the dilatation using single-crystal thermal coefficients ( $\alpha$  and  $\beta$  phase), texture, phase volume fractions, and phase transformation strain.



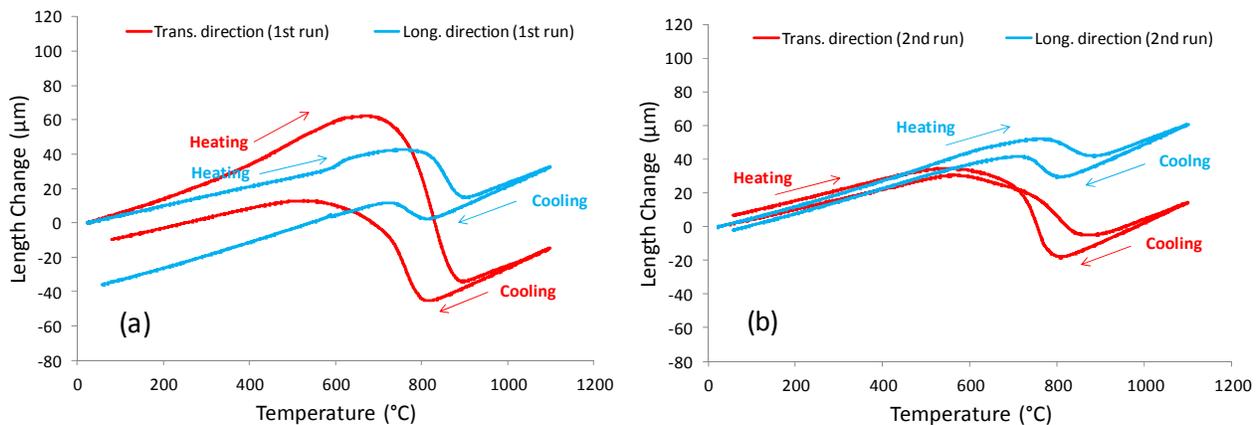
**Figure 2 (a) Photograph showing the test setup in a BÄHR Dilatometer, and (b) same heating and cooling cycle used in dilatation measurements for the 1<sup>st</sup> and 2<sup>nd</sup> run.**

### 3. Results and Analysis

#### 3.1 Heating and Cooling Dilatation Curves

Figure 3 shows the results for the measured dilatations (change in length) plotted as a function heating and cooling temperatures for two consecutive runs using the same pressure-tube sample aligned with the transverse or longitudinal direction of the tube.

In the 1<sup>st</sup> test run (Figure 3a), there are clearly large differences in the length changes observed between the transverse (shown by the red line) and longitudinal samples (shown by the blue line) of an as-manufactured Zr-2.5Nb pressure tube material. During heating to about 600°C, indicated by the red and blue arrows, the transverse sample gave a larger change in length than that in the longitudinal sample. Further heating above 600°C to about 900°C, there is a drastic decrease in the length of these two samples. The dilatational behavior is attributed to the phase transformation of  $\alpha \rightarrow \beta$ , where transformation strains are generated under unconstrained conditions. In the transformation of  $\alpha \rightarrow \beta$ , there is a 10% contraction strain along the  $\langle 11\bar{2}0 \rangle$  directions, and also along two perpendicular directions with a 2% expansion along the  $\langle 0001 \rangle$  directions and a 10% expansion along the  $\langle 11\bar{1}0 \rangle$  directions [6]. The strains associated with the transformation of  $\beta \rightarrow \alpha$  will simply be the reverse of the strains along the three perpendicular directions, respectively. The drastic change in dilatational behavior is observed to be more significant in the transverse sample than in the longitudinal sample. The transverse sample contracted to a large extent (by almost 40  $\mu\text{m}$ ) and the resulting length became shorter than its initial (original) length. For the same temperature heating range (600°C to 900°C), the longitudinal sample's contraction was much less than that in the transverse sample, and it did not shrink to less than its original length.



**Figure 3 Length changes for two heating/cooling runs: (a) Run 1 and (b) Run 2 on the same sample.**

Upon further heating above 900°C to 1100°C, both the transverse and longitudinal samples showed a similar linear expansion characteristics (i.e., slopes are similar). On cooling down to about 800°C after being heated to 1100°C, a similar linear contraction behavior is also seen in these two samples, both showing similar amount of length change (similar slope and extent). On further cooling below 800°C to about 600-700°C, there is a reversal in the dilatational behavior

showing an increasing length change, which means that the sample has expanded (and not contracted during cooling). Further cooling from 600-700°C back to room temperature has resulted in a net shrinkage; the longitudinal sample shortened by more than twice that of the transverse sample. The expansion on cooling down from 800°C to about 600-700°C is attributed to the reversal of the phase transformation ( $\beta \rightarrow \alpha$ ) and the transformation strains that are generated [6]. For the transverse sample, this transition temperature occurred at around 600°C, whereas it is about 700°C for the longitudinal sample.

In Figure 3b, the results for the second consecutive test run are shown for the same transverse and longitudinal samples that were previously tested in the 1<sup>st</sup> thermal cycle. As seen in this Figure 3b, on second heating from room temperature to 500-600°C, there is little difference in the thermal expansion between these two samples. The slopes of the two lines are similar, suggesting that the thermal expansion is nearly isotropic, meaning that the pressure tube sample in the transverse and longitudinal direction of the tube expand to the same extent and in the same manner. This nearly isotropic dilatation behavior is a direct result of some degree of “randomized” texture (Figure 1) generated after the as-manufactured pressure tube sample with its initial (strong) texture (Figure 1) was heated for the first time at 1100°C (1<sup>st</sup> test run) and cooled back down to room temperature. The Kearns’ (texture) numbers [5] listed in Table 1 give an indication of the degree of “randomized” texture. A truly randomized texture will have the Kearns’ number being equal for the three directions of the tube (i.e.,  $f_R = f_T = f_L = 1/3$ ).

As shown in Figure 3b, a further heating from 500-700°C to about 900°C gave a decreasing length change as the pressure tube material goes through another (second) phase transformation from  $\alpha \rightarrow \beta$  with a different amount of transformation strains being produced that is dependent on the starting texture in the previous thermally-cycled sample material. A difference in phase transformation behavior is observed between the transverse and longitudinal samples, with the transverse sample showing a lower transition temperature (i.e.,  $\approx 500^\circ\text{C}$ ) compared to a higher transition temperature of  $\approx 700^\circ\text{C}$  for the longitudinal sample. With continuing heating above 900°C to 1100°C, the two samples gave similar dilatations (i.e., similar length change and slope). On cooling back down to about 800°C, the two samples also showed similar contractions with similar length change and slope. Cooling below 800°C, the two samples started to expand, reversing the contraction that was occurring when the samples were heated from 100°C to just above 800°C. This reversal of dilatation is a direct result of the reversed phase transformation from the  $\beta$ -phase material to an increasing amount of  $\alpha$  phase and the reversed transformation strains that are produced from this phase transformation process [6]. Examination of Figure 3b shows that the phase transformation occurring in the longitudinal sample had completed at a higher temperature of about 700°C compared to the transverse sample that completed its phase transformation at about 600°C. With the final cool down to room temperature, both samples contracted similarly (similar slope) suggesting isotropic dilatation behavior, however the magnitude of the final length change between these two samples was different. The transverse sample experienced a slight net increase in length, whereas the longitudinal sample had a slight net loss in length. Since the magnitude of the net increase and net loss in length change is both small and similar, the dilatation behavior is considered to be almost isotropic, suggesting that a single high-temperature heating and slow cooling back to room temperature (e.g., 1<sup>st</sup> test run)

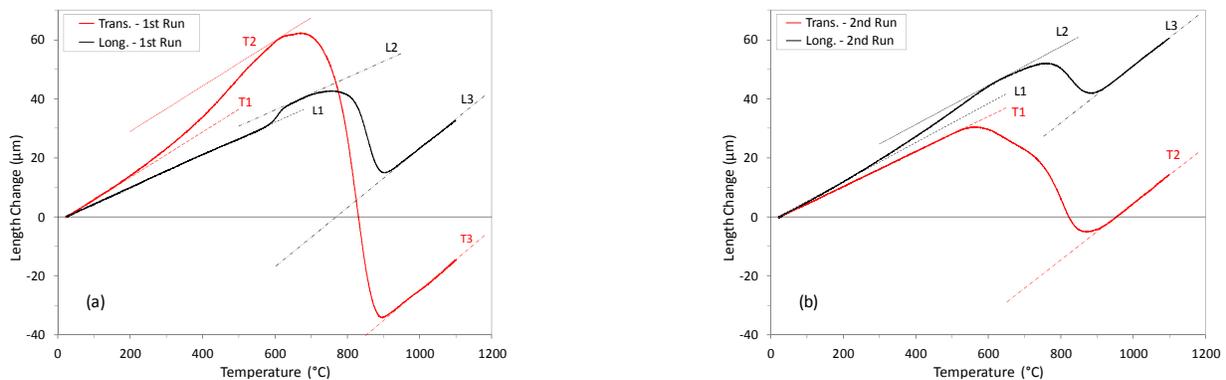
would result in a nearly randomized texture. Such a nearly randomized texture would be similar to that shown in Figure 1.

For the determination of the coefficient of thermal expansion (CTE), only the dilatation curves from the heating portion of the two test runs are used in this analysis as discussed in the following section. The same heating dilatation curves are also used to determine the phase fraction.

### 3.2 Determination of the Coefficient of Thermal Expansion (CTE) and $\beta$ -phase Fraction

Using the heating dilatation curves, the CTE for the low-temperature (below 600°C) and high-temperature (above 900°C) regimes are obtained from the slopes on the curves as indicated with the labels shown in Figure 4.

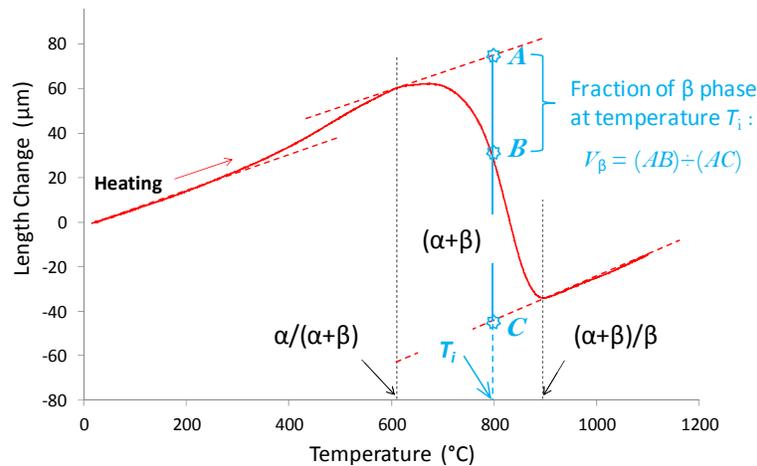
Figure 4a shows the slopes for the 1<sup>st</sup> run (i.e., a first time heating of the as-manufactured pressure tube material). Slopes T1 and T2 correspond to the low-temperature CTE for the transverse sample, and slopes L1 and L2 are for the longitudinal sample. Slope T2 is a parallel offset of slope T1, where the intersection with the curve defines the transition temperature (i.e.,  $\alpha/(\alpha+\beta)$  transus). Similarly, slope L2 is also offset parallel to slope L1, and the intersection point with the curve also defines the transition temperature. The increase in dilatation within the offsets between the two parallel slopes is observed to be significantly more in the transverse sample than in the longitudinal sample. The dilatation increase is likely due to grain shape changes (from highly axially elongated to being equiaxed) along with some grain growth and to a small extent due to increase in the CTE of  $\alpha$ -Zr along the  $\langle c \rangle$  axis before traversing across the  $\alpha/(\alpha+\beta)$  transus. In changing to equiaxed grains during agglomeration, the widths of the elongated grains along the transverse direction would be enlarged, compared to shrinkage of the axial lengths of the grains along the longitudinal direction.



**Figure 4 Length changes for two heating runs: (a) 1<sup>st</sup> run and (b) 2<sup>nd</sup> run on the same sample after completing the 1<sup>st</sup> run. (The slopes labelled “Tx” and “Lx” denote the transverse and longitudinal direction of the pressure tube specimens to determine the CTE values).**

For the high-temperature regime, the CTE of the transverse sample is obtained from the slope T2, and that for the longitudinal sample is taken from the slope L3 (Figure 4b). The tangent points on the dilatation curves before the curve deviates from the slopes T2 and L3 are used to

determine the  $\beta$ -phase fraction within the two phase region. The intersection points (defining the transition temperatures) are used to determine the fraction of  $\beta$ -phase with changes in temperature within the two phase ( $\alpha+\beta$ ) domain. Figure 5 shows a method that is used for estimating the fraction of  $\beta$ -phase material at a particular temperature (e.g.,  $T_i$ ).



**Figure 5 Schematic illustration on determination of  $\beta$ -phase fraction at temperature  $T_i$  using the heating dilatation curve obtained by dilatometry.**

Table 2 lists the CTE results obtained for the low and high temperature regimes for the transverse and longitudinal samples tested with consecutive heating-cooling cycles (Runs 1 and 2). In the 1<sup>st</sup> run of heating and cooling, the transverse sample gave a CTE of about  $7.7 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  compared to the longitudinal sample with about  $5.5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ . The low-temperature anisotropy of thermal expansion is close to 1.4 (a 40% difference), Table 1. In comparison, in the high-temperature regime, the CTE values are about the same ( $10.3$  and  $10.0 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ ) for the transverse and longitudinal samples, giving CTE ratio of 1.03 or nearly unity, which is considered truly isotropic and expected for a bcc  $\beta$ -phase material.

In the 2<sup>nd</sup> run, the low-temperature regime CTE value for the transverse sample is reduced to  $5.9 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ , whereas the longitudinal sample is increased to  $6.6 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ . This CTE ratio of 0.89 (i.e., 11% difference) produced in the 2<sup>nd</sup> run is much smaller than the 40% difference produced in the 1<sup>st</sup> run at the low temperature regime. Heating at the high temperature regime during the 2<sup>nd</sup> run produced a completely isotropic expansion behavior as the CTE ratio is now equal to 1.00 (Table 2) and expected for a bcc  $\beta$ -phase material.

### 3.3 Comparison of $\beta$ -phase Fraction Determined from Dilatometry with Previous Neutron Diffraction Measurements [4]

The evolution of  $\beta$ -phase fraction in the Zr-2.5Nb pressure tube sample as it is heated to a particular temperature within the two phase ( $\alpha+\beta$ ) domain has been analyzed using the method illustrated in Figure 5. The results reported in this study only used the heating dilatation curves for the two consecutive thermal cycles investigated on the same transverse and longitudinal samples.

**Table 2 Thermal expansion coefficients and anisotropy in Zr-2.5Nb pressure tube materials**

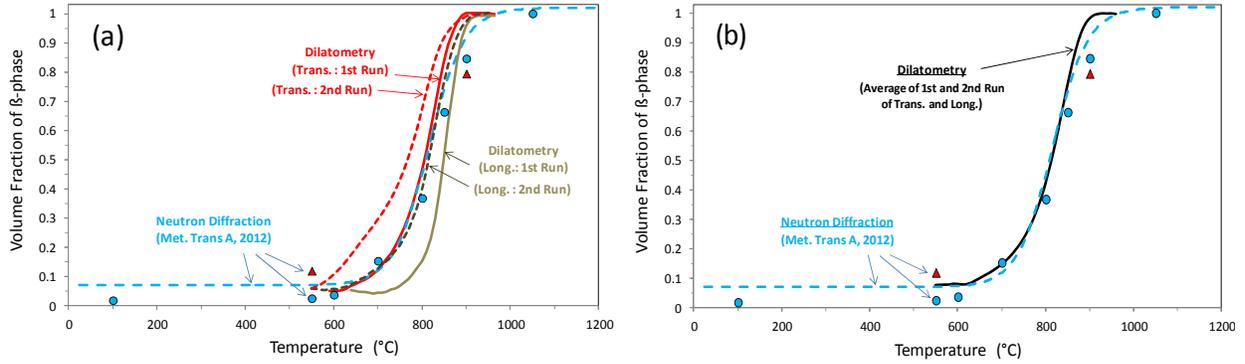
Heating Run No.	Specimen Direction	Coefficient of thermal expansion, CTE ( $\times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ )		CTE anisotropy: $\frac{\alpha_T}{\alpha_L}$	
		Low Temp. (below $\alpha/(\alpha+\beta)$ solvus) (~93% hcp $\alpha$ -phase)	High Temp. (above $(\alpha+\beta)/\beta$ solvus) (100% bcc $\beta$ -phase)	Low Temp. (<600°C)	High Temp. (>900°C)
1 <sup>st</sup>	Transverse	7.7	10.3	1.40	1.03
1 <sup>st</sup>	Longitudinal	5.5	10.0		
2 <sup>nd</sup>	Transverse	5.9	9.6	0.89	1.00
2 <sup>nd</sup>	Longitudinal	6.6	9.6		

Figure 6a gives a summary of the results for the  $\beta$ -phase fraction plotted as a function of temperature. In the same figure, the dilatometry results are compared with previous neutron diffraction measurements [4] of  $\beta$ -phase material in a sample taken from the same parent Zr-2.5Nb pressure tube material that is also used in these dilatometry measurements. In the neutron diffraction experiments, the measurements were essentially made for all orientations of the crystallites of  $\beta$ -phase material in the sample when it was at a thermal equilibrium state (i.e., not measured during a temperature transient). This means that all the  $\beta$ -Zr grains were measured regardless of how they were oriented in the sample (axis) coordinates, thus giving a representative volume fraction of the  $\beta$ -phase material. Examination of Figure 6a shows that the dilatometry results exhibit a large variation from the “equilibrium” results from neutron diffraction [4]. The large variations are largely due to the different starting textures of the  $\alpha$ -Zr grains in the transverse and longitudinal samples, and thus generating a variation in the phase transformation ( $\alpha \rightarrow \beta$ ) strains (discussed in Section 3.2).

In Figure 6b, all the dilatometry curves for the two test runs on the two same samples were averaged to produce one curve. This averaged curve minimizes the effect of texture on the resulting phase transformation strain, and thus gives a quasi-equilibrium fraction for the  $\beta$ -phase material in the Zr-2.5Nb pressure tube sample. The averaged results are in good agreement with the neutron diffraction results [4] (Figure 6b).

### 3.4 Expression for Thermal Expansion using CTE Values of Single Crystals of $\alpha$ - and $\beta$ -phase Zr, Texture, Phase Fraction, and Transformation Strain

Using the  $\beta$ -phase fraction obtained in this dilatometry study and also from previous neutron diffraction measurements [4], expressions have been derived to describe the thermal (dilatation) expansion characteristics of Zr-2Nb pressure tube material. The expressions are derived using the anisotropic CTE values of single crystal of  $\alpha$ -Zr and  $\beta$ -Zr, texture parameters,  $\alpha$  and  $\beta$  phase volume fractions and their proportions, and a net transformation strain associated with a given starting texture in the pressure tube samples.



**Figure 6 Comparison of  $\beta$ -phase fraction between dilatometry and previous neutron measurements [4].**

The semi-empirical expressions are derived for the two principal directions of the pressure tube material, as follows,

Transverse direction:

$$\left(\frac{\Delta L}{L_0}\right)_{Trans} = \left\{ [\alpha_c f_T + \alpha_a (f_R + f_L)] V_\alpha + 1.5 V_\beta \alpha_\beta \left[ \varepsilon_{tx} + 1.5 V_\beta \alpha_\beta \left( \frac{\Delta T - T_1}{T_2} \right) T_3 \right] \right\} \Delta T \quad (1)$$

Longitudinal direction:

$$\left(\frac{\Delta L}{L_0}\right)_{Long} = \left\{ [\alpha_c f_L + \alpha_a (f_R + f_T)] V_\alpha + 1.5 V_\beta \alpha_\beta \left[ \varepsilon_{tx} + 1.5 V_\beta \alpha_\beta \left( \frac{\Delta T - T_1}{T_2} \right) T_3 \right] \right\} \Delta T \quad (2)$$

where,  $\alpha_a$  is the CTE of single crystal of  $\alpha$ -Zr, along the  $a$  axis,  $\alpha_c$  is the coefficient of thermal expansion of single crystal of  $\alpha$ -Zr along the  $c$  axis.  $\alpha_c = 11.8 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  and  $\alpha_a = 5.19 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  are averaged values from 25 $^\circ\text{C}$  to 760 $^\circ\text{C}$  [6] where  $\alpha_a$  is a constant and  $\alpha_c$  increase with temperature.  $V_\alpha = (1 - V_\beta)$  is the volume fraction of  $\alpha$ -phase material.  $\alpha_\beta = 3.14 \times 10^{-5} \text{ }^\circ\text{C}^{-1}$  is the coefficient of thermal expansion of the  $\beta$ -phase material [7].  $V_\beta$  is the volume fraction of  $\beta$ -phase material given by  $V_\beta = 0.07 + 0.95 / (1 + \exp(-(T - 820^\circ\text{C})/40^\circ\text{C}))$ .  $f_T, f_R,$  and  $f_L$  are Kearns' (texture) numbers for the resolved fractions of basal plane normals in the principle axes of the pressure tube samples.  $\Delta T = (T_{heating} - T_{RT})$  is temperature difference between heating temperature ( $T_{heating}$ ) and room temperature ( $T_{RT}$ ).  $T_1 = 760^\circ\text{C}$ ,  $T_2 = 600^\circ\text{C}$  ( $\approx \alpha/(\alpha+\beta)$  transus),  $T_3 = 1860^\circ\text{C}$  ( $\approx$  liquidus temperature of Zr).  $\varepsilon_{tx}$  is the transformation strain associated with the phase transformation of  $\alpha \rightarrow \beta$  estimated for the starting crystallographic texture of the  $\alpha$ -Zr grains in the pressure tube samples.

Table 3 lists an estimated transformation strain for the samples with different starting textures (Figure 1), where a net strain is assumed to be associated with the direction of the dilatation measurements on the samples (discussed in Section 3.2). This net transformation strain is assumed on the basis of equal participation of all (hcp)  $\alpha$ -Zr grains to be capable of transforming to the (bcc)  $\beta$ -Zr phase structure during the transient heating employed in the dilatometry measurements.

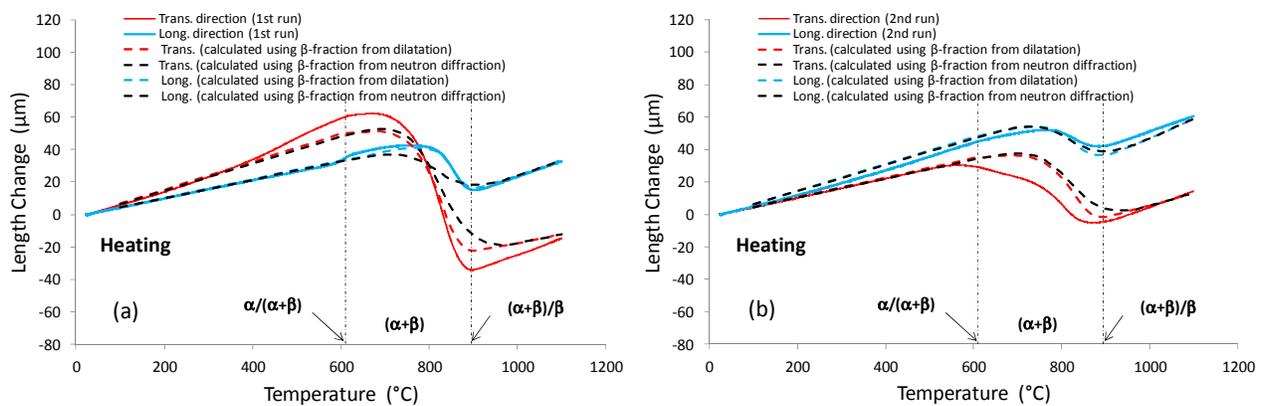
**Table 3 Transformation strain ( $\epsilon_{tx}$ ) estimated for transformation of  $\alpha \rightarrow \beta$  on heating.**

$\epsilon_{tx}$	Transverse	Longitudinal
Run 1	-0.07	0.02
Run 2	-0.02	0.07
Randomized (averaged over all runs and directions)	0.0	

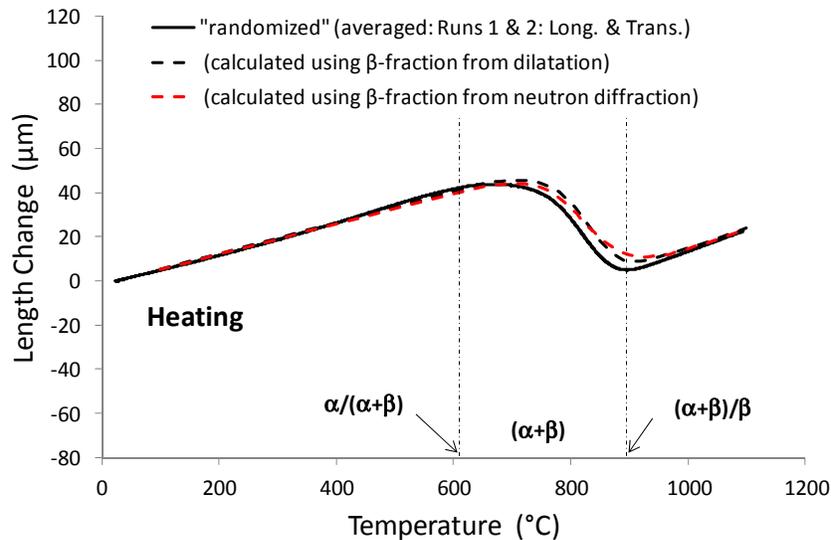
Figure 7a shows a comparison of the calculated results with the dilatation measurements from the 1<sup>st</sup> test run. The comparison for the 2<sup>nd</sup> test run is shown in Figure 7b. Examination of the 1<sup>st</sup> test run (comparison shown in Figure 7a) suggests that the calculated results are consistent with measurements with good agreement in the low-temperature regime (below the  $\alpha/(\alpha+\beta)$  solvus). In the two-phase ( $\alpha+\beta$ ) domain, the agreement is not so good despite accounting for the effect of phase transformation.

Examination of Figure 7b indicates that the comparison between the calculations and the dilatation curves has improved. The transverse sample showed agreement for the full range of temperatures. For the longitudinal sample, however, good agreement is only seen at the low and high ends of the temperature range; in the two-phase domain, calculations had over predicted the change in length during heating.

A further calculation of the dilatations was obtained by taking a randomized texture. With a randomized texture, the transformation strain becomes effectively zero (Table 3). A comparison of the randomized-texture calculations with the averaged dilatation curves showed good agreement (Figure 8).



**Figure 7 Comparison of thermal expansion of as-manufactured Zr-2.5Nb pressure tube material (Run 1 and 2) between measured and the calculated values obtained using phase fractions from: (a) dilatation measurements (this study), and (b) previously measured neutron diffraction [4].**



**Figure 8 Comparison of thermal expansion between calculated and measurements (averaged) of two runs for transverse and longitudinal samples of Zr-2.5Nb pressure tube.**

#### 4. Concluding Remarks

The following conclusions can be summarized from this dilatometry study.

1. The first heating of an as-manufactured Zr-2.5Nb pressure tube material to 1100°C has shown that the material expand significantly different between the transverse and longitudinal samples of the pressure tube. At low temperature (below 600°C), the thermal expansion anisotropy is about 40%, with transverse sample expanding much more than the longitudinal sample when heated at the same rate to reach the same temperature. This expansion anisotropy is largely due to the strong transverse texture of  $\alpha$ -Zr grains in the as-manufactured pressure tube starting material.
2. Heating the transverse and longitudinal samples at high temperatures (above 900°C) within the  $\beta$ -phase domain produced nearly isotropic expansion behavior, with only about 3% difference in their coefficients of thermal expansion. Such an isotropic behavior is expected for a bcc-structured  $\beta$ -Zr material which remains stable at high temperatures.
3. Heating at 600°C to 900°C, traversing the two phase ( $\alpha+\beta$ ) domain resulted in vastly different dilatation behavior for both the transverse and longitudinal samples. The transverse sample showed a large contraction with increasing temperatures ending up being shorter than its initial (original) length (i.e., it shrunk significantly). In comparison, the dilatation behavior of the longitudinal sample was not so drastic. It also got shorter with increasing temperatures without ending up being shorter than it original length. The thermal expansion anisotropy persisted through the two phase region, a direct consequence of the initial strong texture of the  $\alpha$ -Zr material in the pressure tube samples. Essentially the pressure tube material contracts during heating as the temperatures traverse across the two phase ( $\alpha+\beta$ ) region.

4. On subsequent heating of the same two samples from room temperature after being first heated at 1100°C, the starting microstructures (texture and grain structure) have been modified from its initial state before they were heated. At the low temperatures (below 600°C), the transverse and longitudinal samples showed a reduction in the anisotropy of thermal expansion to about 11%. Heating through the two phase region (i.e., 600°C to 900°), the dilatation behavior between the two samples has also significant differences. Above 900°C, isotropic dilatation behavior between the two samples was observed for the bcc  $\beta$ -Zr material.
5. Semi-empirical dilatation expressions have been derived that included microstructural parameters for  $\beta$ -phase fraction from this dilatometry study and previous neutron diffraction measurements [4], thermal expansion coefficients of single crystal of  $\alpha$ -Zr and  $\beta$ -Zr, Kearns' (texture) numbers, phase fraction and transformation strain for  $\alpha \rightarrow \beta$ . Comparisons using a "randomized" starting texture where a net transformation strain is estimated to be zero, gave good agreement between the calculations and the averaged ("randomized") dilatation curves for the two samples and test runs.

## 5. References

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