

# Isothermal stress reorientation of hydrides in Zr-2.5Nb pressure tube alloy



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## ABSTRACT

The precipitation of hydrides normal to the transverse direction in Zr-2.5Nb pressure tube alloy when cooled under stress above a threshold value, is known as stress reorientation of hydrides. The ex-situ threshold stress values reported in the literature were determined using pre-charged samples heated to a peak temperature to dissolve all the hydrides, cooled to reorientation temperature followed by cooling under stress from reorientation temperature. Leger and Donner performed in-situ reorientation experiments using samples having hydride deposited on its surface. In both cases, hydride reorientation occurred during cooling. In the present investigation, for the first time, experiments were done to study the reorientation phenomenon using in-situ gaseous charging of samples under stress at a constant temperature using a specially designed set up. It is expected that the threshold stress determined by in-situ method used in the present investigation will more closely resemble the formation of radial hydrides under reactor operating conditions. In this work, the threshold stress values for reorientation of hydrides in Zr-2.5Nb pressure tube material were determined using both in-situ and ex-situ methods.

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## 1. Introduction

Zr-2.5Nb alloy is used as pressure tube material in pressurized heavy water reactors (PHWR) due to its low neutron absorption, good corrosion resistance and adequate high temperature strength [1,2]. Hydride embrittlement degrades the mechanical properties of Zr-2.5Nb pressure tube (PT) material, which may limit the life of the pressure tubes in PHWR [3,4].

The initial hydrogen content of PT is kept as low as possible during the manufacturing process of pressure tubes [5,6] to ensure high initial fracture toughness. However, hydrogen can be picked up during service. Hydrogen present in excess of terminal solid solubility precipitates as hydrides [3,7,8] of plate shaped morphology. The orientation of hydrides depends on the texture, microstructure of the PT and stress (applied as well as residual) during its precipitation. The degree of embrittlement depends on hydrogen concentration, hydride volume fraction, size, distribution, and orientation [3,9]. The texture of the pressure tube is such that there are two possible orientations of hydride. The hydrides oriented along the circumferential–axial plane are called circumferential hydrides and those oriented along the radial–axial plane are called radial

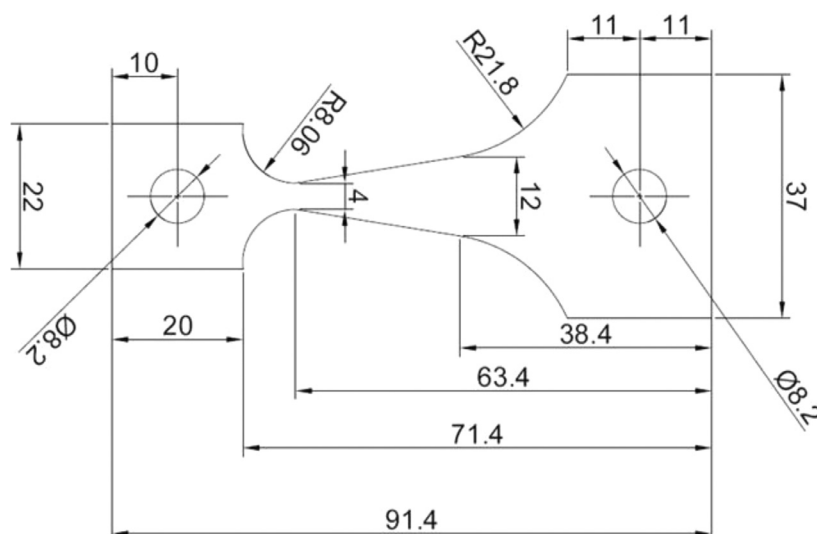
hydrides. Due to the microstructure of pressure tubes, only circumferential hydrides form in unstressed condition. However, radial hydrides normal to transverse direction can be formed when cooled under stress. The phenomenon of formation of radial hydride when cooled under stress, is called as stress-reorientation of hydride and it is associated with a threshold value ( $\sigma_{th}$ ) [9,10].

Hardie and Shanahan [11] and Singh et al. [9,10] determined the threshold stress value experimentally using tapered gage hydrogen pre-charged samples obtained from Zr-2.5Nb alloy pressure tube material. They subjected the samples to reorientation heat treatment in which samples were heated to a pre-determined solution annealing temperature ( $T_A$ ) and cooled from solution annealing temperature to reorientation temperature ( $T_R$ ), soaked at reorientation temperature, and finally, samples were cooled under load from the reorientation temperature to ambient temperature to facilitate reorientation of hydrides. Throughout this manuscript, experiments in which samples were subjected to the above heat treatment are referred to as ex-situ reorientation tests. Colas et al. [12] studied the mechanism of hydride reorientation in pre-charged Zircaloy-4 samples subjected to thermal cycles using synchrotron X-Ray diffraction analysis.

In order to remove the ‘memory effects’ [13] due to pre-existing hydrides on reorientation phenomenon, Leger and Donner [14] had done reorientation experiments in which hydrogen was introduced

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**Fig. 1.** Sketch of tapered gage tensile sample used in the present investigation to determine the threshold stress (All dimensions are given in mm) [9,10].

into the sample under stress by electrolytically depositing hydride on surfaces of the sample. Upon heating, hydrogen ingressed into the material from the surface hydride layer till it attained a bulk hydrogen concentration equal to the terminal solid solubility for dissolution (TSSD). Thermodynamically, the maximum amount of hydrogen that can be charged into the sample in this method is TSSD at the annealing temperature. Therefore, hydride reorientation cannot occur at a constant temperature. Effectively, hydride reorientation occurred during the cooling at a temperature less than reorientation temperature ( $T_{R1}$ ) dictated by the temperature dependence of terminal solid solubility for precipitation (TSSP).

In the experiments reported in this article, samples were charged using a gaseous charging technique using Sievert's apparatus [15] and for the first time, experiments were done to study the reorientation phenomenon using in-situ gaseous charging of samples under stress at a constant temperature using a specially designed set up. In this article threshold stress for the reorientation of hydrides in Zr-2.5%Nb alloy pressure tube material was determined at 573 K using in-situ stress reorientation test and compared with ex-situ stress reorientation test results.

## 2. Material and methods

Zr-2.5%Nb alloy pressure tubes of PHWR 220 MWe manufactured employing double radial forging followed by the single pilgering, were received from the NFC Hyderabad [2] and these tubes were cut into spools of length 100 mm. The spools were axially slit and warm pressed at 673 K, followed by cold rolling to get the flat plates. The flattened plates were subjected to stress relieving at 673 K for 24 h. Tensile specimens with tapered gage and with specimen axis along the circumferential direction of the tube were fabricated as per the drawing shown in Fig. 1 [9,10]. These specimens were polished up to 800 grit silicon carbide abrasive papers to remove the autoclaving layer and to get a fresh, contamination free surface for hydrogen charging.

### 2.1. Ex-situ stress reorientation treatment

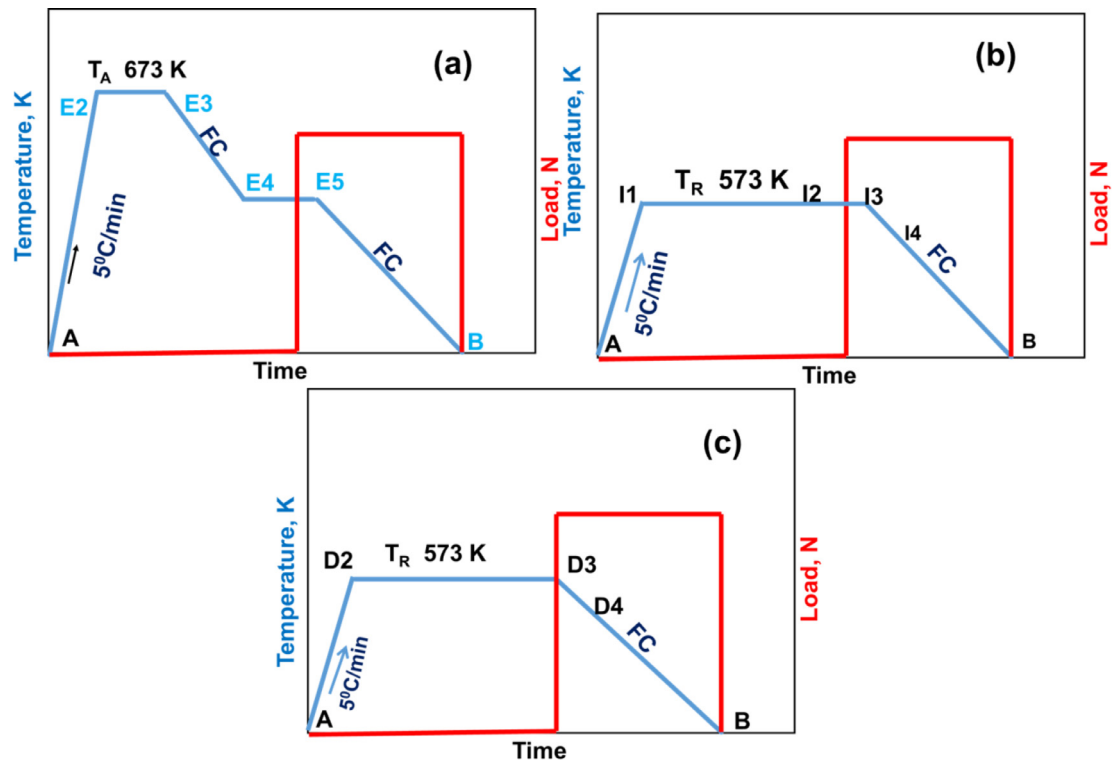
The polished tapered gage samples were charged with 95 wppm of hydrogen using a gaseous hydrogen technique [15]. The samples were subjected to homogenization treatment at 673 K for 24 h and furnace cooled to room temperature to get the uniform

distribution of hydrides in the sample. The hydrogen charged samples were loaded in the constant load creep testing machine. The samples were subjected to reorientation heat treatment at 573 K. The reorientation heat treatment at 573 K consists of heating the sample to 673 K and holding at this temperature for 60 min to take all the hydride in solution and annihilate the dislocation networks formed around the hydride precipitates, furnace cooling the samples to reorientation temperature, and soaking at this temperature for 45 min to attain thermal equilibrium, followed by cooling the sample to ambient temperature under stress to reorient the hydrides. For all the tests, samples were furnace cooled and three thermocouples were mounted at lower shoulder, upper shoulder, and middle of the sample to ensure the uniform temperature all along the sample. The schematic of thermal cycle {A-E2-E3-E4-E5-B} followed for ex-situ reorientation treatment is shown in Fig. 2(a).

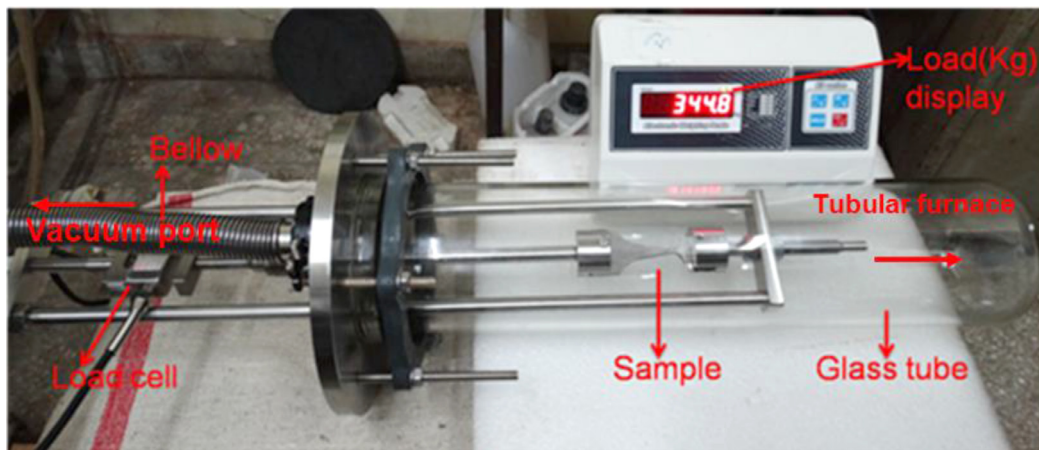
### 2.2. In-situ stress reorientation treatment

#### 2.2.1. In-situ loading and charging setup

The polished samples were loaded into a specially designed setup in which samples can be charged with hydrogen under stress at a constant temperature. The details of the setup are given in Fig. 3. As shown in Fig. 3, the setup consists of a load train in which sample, pull rod and load cell are connected in a series. The load train is supported by two rods, which are welded to a flange. The pull rod is connected to the flange through a brass bearing. A flexible bellow is welded to flange and pull rod to accommodate the displacement of pull rod, and to ensure vacuum leak tightness. The samples can be loaded to the required load by rotating the nut. The load cell with its display unit is connected to the load train to measure and record the applied load. Ports are provided in the flange to measure the temperature of the sample and to connect system to the vacuum pump. The entire setup after sample loading is put into the glass tube and inserted into a resistance heated furnace. The hydrogen is released into the glass tube after achieving the required vacuum. Subsequently, the system is isolated and heated to reorientation temperature using the furnace. The sample is kept under load at constant temperature till the required amount of hydrogen is charged in it. The thermal cycle {A-I1-I2-I3-I4-B} used for in-situ stress reorientation is illustrated in Fig. 2(b).



**Fig. 2.** Schematic of thermal cycles followed in (a) ex-situ stress reorientation (A-E2-E3-E4-E5-B), (b) in-situ reorientation (A-I1-I2-I3-I4-B) and (c) Leger and Donner [14] (A-D2-D3-D4-B).  $T_A$  –solution annealing temperature,  $T_R$  – reorientation temperature, FC- Furnace Cooling.



**Fig. 3.** The specially designed set up used for the in-situ stress reorientation treatment.

The schematic of thermal cycle {A-D2-D3-D4-B} followed by Leger and Donner [14] is shown in Fig. 2(c). The samples were heated to 573 K and held for 48 h and load was applied during cooling. The maximum amount of hydrogen that can be charged during this thermal cycle is TSSD.

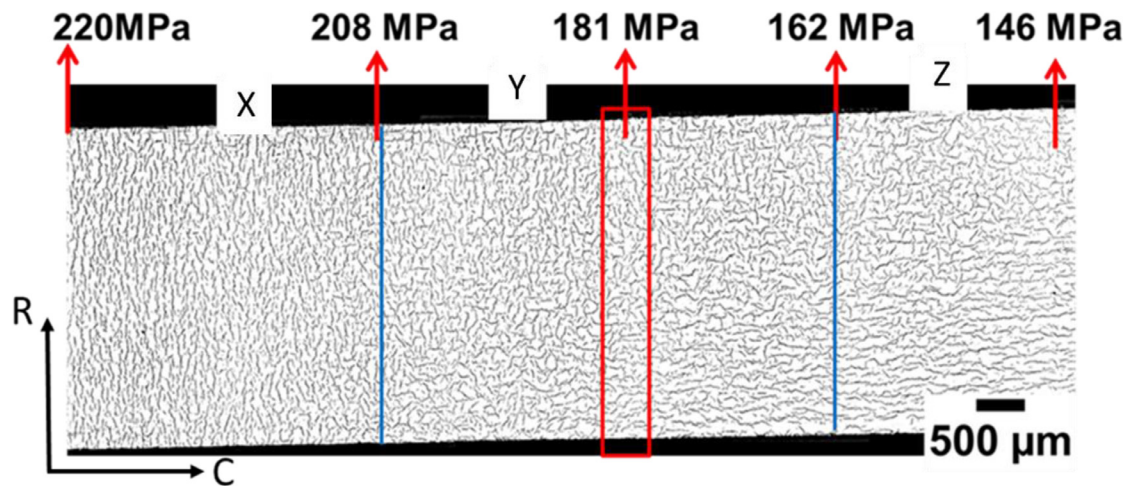
### 2.3. Metallography

The tested specimens were sectioned along the radial-circumferential plane, ground using 800 grit silicon carbide abrasive papers followed by swab etching with the solution of HF: HNO<sub>3</sub>: H<sub>2</sub>O: 10: 45: 45 for about 15 s. A montage of optical images was prepared and stress variation along the cross-section was calculated and threshold stress for reorientation was determined using the half thickness method [9,10,14].

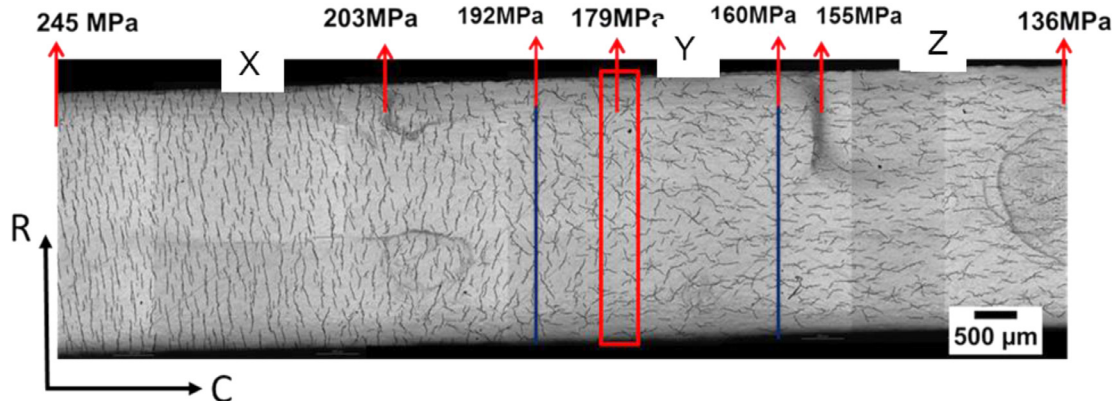
## 3. Results

### 3.1. Reorientation stress, $\sigma_{th}$

Duplicate tests were performed using tapered gage samples to determine the threshold stress values at 573 K. Both the values were within 3%. These threshold stress values were verified by performing stress reorientation experiments using parallel gage samples. The montages of hydride micrographs on radial-circumferential plane showing the corresponding stress variation across the gage length for both ex-situ stress reorientation heat treatment and in-situ stress reorientation is shown in Fig. 4 and Fig. 5, respectively. As shown in Figs. 4 and 5, the hydride montage can be divided into three distinct regions, namely, 'region X' in which fully radial hydrides formed, 'region Y' in which mixed



**Fig. 4.** Montage of the hydride micrographs showing the variation in hydride orientation across the gage length of tapered gage tensile specimen subjected to ex-situ reorientation treatment at 573 K. The numbers above the montage represent the externally applied stress in MPa during the reorientation treatment. The montage is demarcated into three regions (X – fully radial, Y - mixed, Z- fully circumferential) based on the orientation of hydrides. The region containing 50% radial hydrides across the thickness is marked red color box (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article).



**Fig. 5.** Montage of the hydride micrographs showing the variation in hydride orientation across the gage length of tapered gage tensile specimen subjected to in-situ reorientation treatment at 573 K. The numbers above the montage represent the externally applied stress in MPa during the reorientation treatment. The montage is demarcated into three regions (X – fully radial, Y - mixed, Z- fully circumferential) based on the orientation of hydrides. The region containing 50% radial hydrides across the thickness is marked red color box (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article).

hydrides formed, and ‘region Z’ in which circumferential hydrides formed. The percentage of thickness in which radial hydrides in the region Y is observed to be decreasing with decrease in stress from 208 MPa and 162 MPa is shown in Fig. 4. The stress corresponding to cross-section in which a minimum of 50% of thickness contains radial hydrides (half thickness method) [9,10,14] is considered as threshold stress ( $\sigma_{th}$ ) for reorientation of hydrides. It is found that the threshold stress ( $\sigma_{th}$ ) for reorientation in case of ex-situ reorientation treatment is 181 MPa. Similarly, a montage of hydride micrograph was prepared for the samples subjected to in-situ stress reorientation treatment and mixed hydride orientation observed in the ‘region Y’ with corresponding stress values ranging from 192 MPa to 160 MPa is shown in Fig. 5. The threshold stress ( $\sigma_{th}$ ) in the case of samples subjected to in-situ stress reorientation treatment at 573 K is found to be 179 MPa, which is very much close to the value determined using ex-situ stress reorientation treatment.

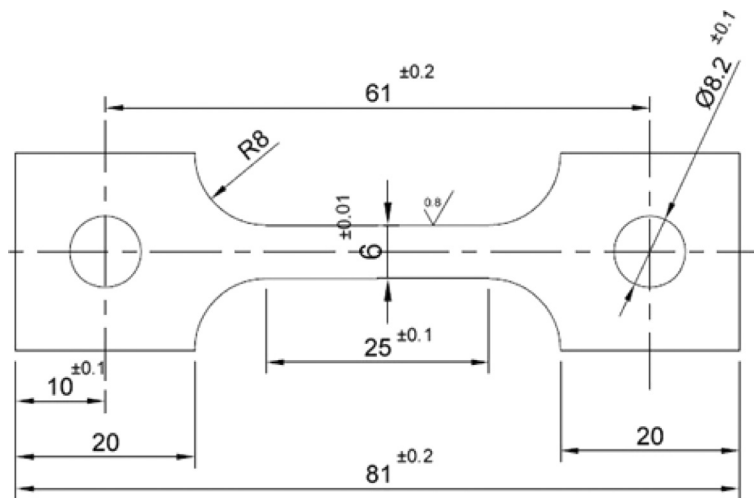
### 3.2. Validation of reorientation stress, $\sigma_{th}$

To validate the reorientation stresses determined using tapered gage samples, standard tensile samples with 25 mm gage length

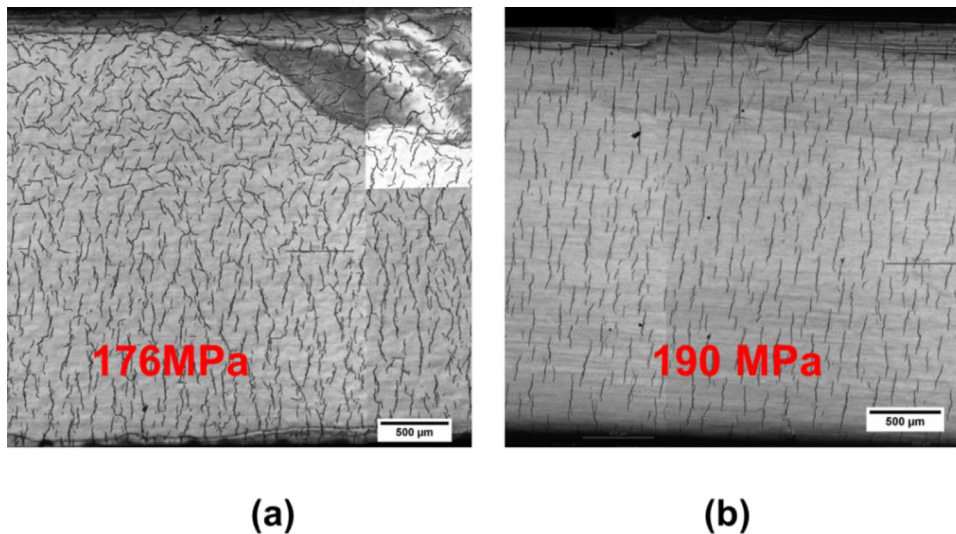
and 6 mm width as shown in Fig. 6, were fabricated from the same plates and pre charged with 95 wppm hydrogen. The parallel gage samples were subjected to ex-situ reorientation treatment at 573 K (as per the thermal cycle shown in Fig. 2a) under stress values of 176 MPa and 190 MPa (stresses corresponding to ‘region Y’ as shown in the Figs. 4 & 5). The samples were metallographically examined and hydride micrographs are shown in Fig. 7. It can be observed that mixed hydrides having radial hydride fraction more than 50% of thickness are formed for the sample subjected to reorientation treatment under stress value of 176 MPa (Fig. 7a) whereas, through thickness radial hydrides are observed for sample subjected to reorientation treatment under stress value of 190 MPa (Fig. 7b).

## 4. Discussion

It is reported that the precipitation of hydride platelets involves three stages i.e. nucleation, precipitation, and coarsening [16–18]. The phenomenon of hydride reorientation is more pronounced in the nucleation stage and it also depends on the thermal history prior to cooling and the existence of circumferential hydrides while cooling. The hydride precipitation is associated with memory effect



**Fig. 6.** Sketch of parallel gage tensile sample used in the present investigation to validate the threshold stress determined using tapered gage tensile samples.



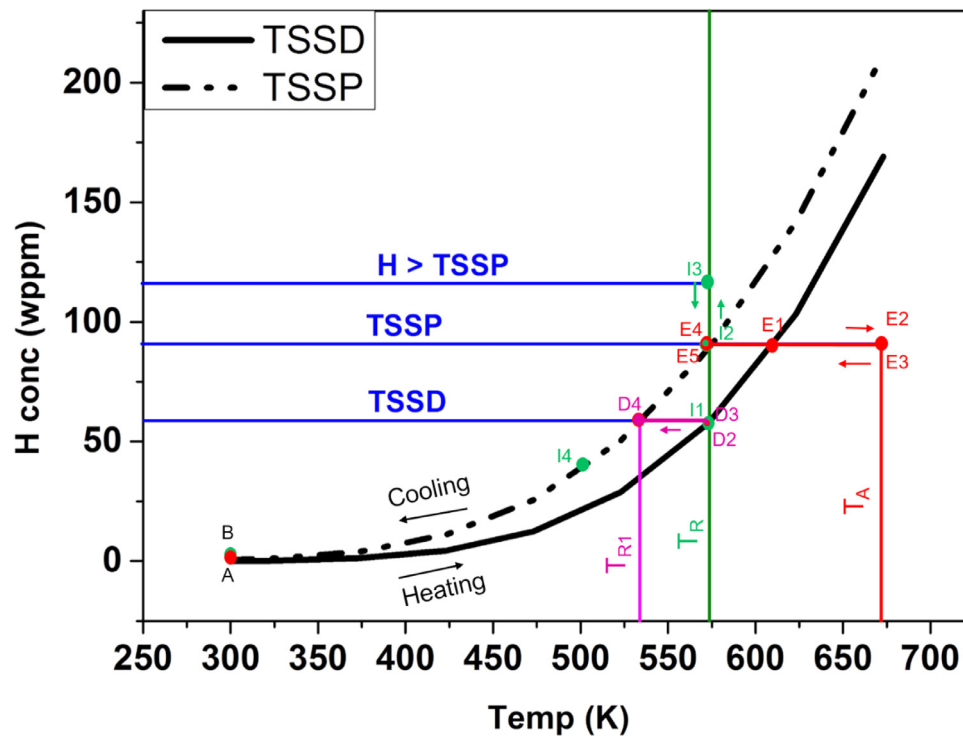
**Fig. 7.** Optical micrographs showing the orientation of hydrides on RC plane of tensile gage sample subjected to ex-situ reorientation treatment at 573 K under (a) 176 MPa (b) 190 MPa.

in which dislocation networks formed during the precipitation acts as nucleation sites for re-precipitation of hydrides. The threshold stress is expected to be higher in the presence of the pre-existing circumferential hydrides or the dislocation network present at pre-existing hydride locations as the nucleation of radial hydrides has to compete with the growth of the pre-existing circumferential hydride or the nucleation of circumferential hydride over pre-existing circumferential locations.

Thus the precipitation of radial hydrides depends on the initial hydrogen concentration and the presence of pre-existing circumferential hydrides. If the solution annealing temperature is less than certain values such that complete dissolution of hydride doesn't take place, either pre-existing hydrides or dislocation network will favor the growth or nucleation of circumferential hydrides rather than nucleation of radial hydrides. Therefore, threshold stresses in such cases will be higher when compared to the cases in which samples are subjected to solution annealing temperature such that hydrides are completely dissolved and the dislocation network is annihilated.

#### 4.1. Ex-situ reorientation

It is reported that TSSP depends on the maximum temperature ( $T_{max}$ ) of the thermal cycle and TSSP decreases with an increase in the  $T_{max}$  [7,8]. For  $T_{max}$  above a critical value, which depends on hydrogen concentration, TSSP becomes independent of  $T_{max}$ . The difference in the TSSP is attributed to the annihilation of the dislocation network formed around the hydrides during its precipitation. The thermal cycle {A-E2-E3-E4-E5-B} used for ex-situ reorientation treatment in the present work is mapped onto the TSS curves of Zr-2.5Nb alloy and is shown in Fig. 8. The pre-charged samples with 95 wppm hydrogen were heated to 673 K (point E2 in Fig. 8) and held for 60 min to completely dissolve hydrides and to annihilate the dislocation network formed around the hydrides [9,10]. This was followed by furnace cooling the samples to the reorientation temperature of 573 K (point E5 in Fig. 8) and the load was applied to the sample. Since the partial molar volume of hydrogen in  $\alpha$ -Zr and  $\delta$ -hydride is nearly same [11,18] the applied stress influences neither dissolution nor precipitation of hydrides.



**Fig. 8.** The mapping of thermal cycles of ex-situ stress reorientation {A- E1-E2-E3-E4-E5-B}, Leger and Donner{A-D2-D3-D4-B}[14] and in-situ stress reorientation {A-I1-I2-I3-I4-B} on to the TSS curves of Zr-2.5Nb pressure tube alloy [7].

Thus slight undercooling to a temperature where hydrogen concentration in the sample is more than the TSSP is necessary for the precipitation of hydrides. If the applied stress is more than the threshold stress radial will nucleate and continue to grow with further cooling.

#### 4.2. In-situ reorientation

The thermal cycle {A-D2-D3-D4-B} followed by Leger and Donner [14] for stress reorientation treatment is also mapped onto the TSS curves of Zr-2.5Nb alloy as shown in Fig. 8. Upon heating the sample with hydride layer on its surface, the maximum hydrogen that can be dissolved into the sample from the hydride layer is TSSD, which is about 50–55 wppm of hydrogen at 573 K as can be seen from Fig.8 (point D3). However, TSSP for precipitation of hydride at 573 K is around 90–100 wppm of hydrogen. Therefore, hydrides cannot be precipitated at 573 K in the samples containing 50–55 wppm of hydrogen. Instead, they start to precipitate at the temperatures lower than the test temperature ( $T_{R1}$ ) as shown in Fig. 8 (point D4). Thus the reorientation temperature reported by Leger and Donner[14] corresponds to temperature D4 in Fig. 8 and not 573 K.

In the case of in-situ reorientation treatment performed in the present work, hydrogen is gaseously charged under stress at a constant temperature. Hence, there is no history of preexisting hydrides i.e. no prior preferable nucleation sites for hydride platelet formation. The novelty of the present in-situ experiments is that hydrogen in excess of TSSD can be charged in the present setup, thereby precipitation of hydrides can take place at a constant temperature. The thermal cycle {A-I1-I2-I3-I4-B} of in-situ reorientation treatment is also mapped onto the TSS curves of Zr-2.5Nb alloy as shown in Fig. 8. Upon continuous charging of hydrogen at a constant temperature, nucleation sites for radial hydrides will be generated whenever hydrogen concentration exceeds TSSP and stress is greater than the threshold value. With further in-

crease in hydrogen concentration, radial hydrides will grow under isothermal condition. The threshold thus determined will be under isothermal conditions, which will be close to the formation of radial hydride in pressure tubes under reactor operating conditions.

The threshold stress for the reorientation of hydrides is reported to be strongly dependent on the strength of the material [9]. However, the strength of Zr-2.5%Nb shows a weak dependence on the temperature between 523 and 573 K [19]. Therefore, one can expect no difference in threshold values obtained using in-situ stress reorientation treatment and ex situ heat treatment with sufficient solution annealing temperature to dissolve all the preexisting hydrides and annihilate the dislocation network around the hydrides.

#### 5. Summary and conclusions

The threshold stress for reorientation of hydrides in cold worked and stress relieved Zr-2.5Nb alloy pressure tube material was determined using tapered gage tensile samples subjected to both ex-situ and in-situ reorientation treatments at 573 K and validated using parallel gage samples. For the first time, the in-situ stress reorientation treatment was carried out using a specially designed set up, which allowed in-situ gaseous hydrogen charging of samples under stress at a constant temperature. The threshold stress values at 573 K determined using both in-situ and ex-situ reorientation treatments were found to be comparable, provided the ex-situ stress reorientation treatment is carried out by cooling the samples from a maximum temperature more than a critical value.

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## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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