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Stress-reorientation of hydrides and hydride embrittlement of Zr–2.5 wt% Nb pressure tube alloy ☆

R.N. Singh [a,*], R. Kishore [a], S.S. Singh [b], T.K. Sinha [a], B.P. Kashyap [c]

[a] *Materials Science Division, Bhabha Atomic Research Centre, Mumbai 400 085, India*
[b] *Summer project trainee from Department of Metallurgy & Materials Engineering, IIT Roorkee, Roorkee 247 667, UA, India*
[c] *Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology, Powai, Mumbai 400 076, India*

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Abstract

Hydrogen in excess of the terminal solid solubility precipitates out as a brittle hydride phase in zirconium alloys. The hydrides acquire platelet shaped morphology due to their accommodation in the matrix and can cause severe embrittlement, especially when these are oriented normal to the tensile stress axis. The precipitation of hydride platelets normal to the tensile stress when cooled under stress from a solution-annealing temperature is commonly referred to as 'stress-reorientation'. The stress-reorientation is associated with a threshold stress below which no reorientation is observed. In this work, stress-reorientation of hydrides was investigated for unirradiated, cold worked and stress-relieved Zr–2.5 wt% Nb pressure tube material for a reorientation temperature of 423–723 K. The effect of the reoriented hydrides on the tensile properties of the Zr–2.5 wt% Nb pressure tube alloy was evaluated in the temperature range of 298–573 K.

1. Introduction

Apart from satisfying the generic requirements of the structural materials like good corrosion resistance and adequate high temperature strength and ductility under operating conditions, dilute Zr-alloys meet the specific requirements of the nuclear structural materials (low neutron absorption cross-section, and microstructural and irradiation stability) required for application as core structural components in water-cooled nuclear reactors [1–7]. Tubes made from Zr–2.5 wt% Nb alloy [1–7] in cold worked and stress-relieved (CWSR) condition, are called pressure tubes, which act as miniature pressure vessels in Pressurized Heavy Water Reactors (PHWR) and operate over the temperature range of 526–566 K under an internal pressure of ∼10.5 MPa, and neutron flux of the order of 3×10^{17} n/m^2/s. The initial hydrogen content of the pressure tubes is kept as low as possible by controlling the manufacturing process parameters [8]. However, part of the hydrogen/deuterium evolved during service from coolant-metal corrosion reaction is picked up by the pressure tubes of the PHWR. Hydrogen present in excess of terminal solid solubility (TSS) [6] precipitates out as hydride phase. Depending upon the hydrogen content, cooling rate and temperature of hydride precipitation, either of the three γ-, δ- or ε-hydrides can form in dilute zirconium alloys [6]. However, under the cooling rate possible for the pressure tubes under reactor operating conditions, only δ-hydride precipitate of platelet morphology is known to form [6] in the operating pressure tubes. Being brittle, the presence of substantial quantities of hydrides can cause embrittlement of the host matrix resulting in loss of ductility, impact and fracture toughness. However, for a significant reduction in these properties, a certain minimum volume fraction of the embrittling phase [9–11] is required and

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* Corresponding author. Fax: +91-22 5505239.
E-mail address: mms@apsara.barc.ernet.in (R.N. Singh).

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degree of embrittlement is strongly influenced by the orientation of hydride platelets. The factors reported to affect δ-hydride platelet orientation in Zr alloy are crystallographic texture, prior strain and applied stress [6,12]. Hydride precipitation under stress shows preference for the orientation of hydride, where platelet normal is parallel to tensile stress direction [6,13–18] and this phenomenon is called stress-reorientation.

The texture of the CWSR Zr–2.5 wt% Nb alloy is such that crystallographically, only two orientations are permissible, viz., along the circumferential–axial and the radial–axial planes [6], which are respectively called circumferential and radial hydrides, as illustrated schematically in Fig. 1. The microstructure of the CWSR Zr–2.5 wt% Nb alloy pressure tube material is such that under unstressed condition, only circumferential hydrides form [6,17]. For these components, mode of stress-reorientation of hydrides is the precipitation of radial hydrides, while cooling under stress from a solution-annealing temperature [6,9–21]. This is associated with a critical stress called threshold stress for reorientation below which no reorientation occurs [6,17,18]. The threshold stress is reported [6] to increase with increase in material strength and decrease in solution-annealing temperature. It is suggested that the threshold stress for reorientation of hydrides in CWSR Zr–2.5 wt% Nb pressure tube alloy reduces to 0 at a temperature of 704 K [18]. A high concentration of basal poles parallel to the applied stress is necessary for reorientation of hydrides [6,20,21]. However, the degree of reorientation is reported to be insensitive to time under stress; and also small variation in texture, grain-size and dislocation density are found to have apparently no consistent effects [6].

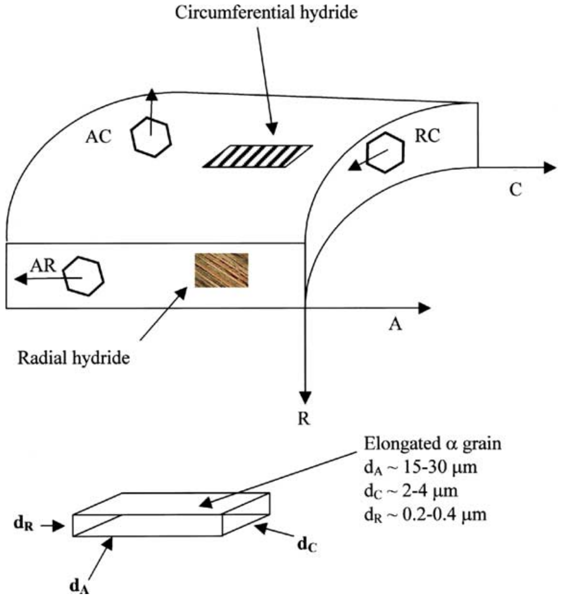

Fig. 1. A section of pressure tube illustrating the orientation of circumferential and radial hydrides [6] is shown schematically. Typical α phase grain dimensions [24] observed in cold worked and stress-relieved Zr–2.5 wt% Nb pressure tube alloy. A – axial, C – circumferential and R – radial directions. AR – axial–radial, RC – radial–circumferential and AC – axial–circumferential planes are also indicated.

A literature survey [6,18] show that the temperature dependence of the threshold stress for reorientation of hydrides in CWSR Zr–2.5 wt% Nb alloy is yet to be clearly established. Also an understanding of the stress-reorientation behavior of hydrides will help in understanding the hydride precipitation and its preferred orientation during delayed hydride cracking [22] and hydride blister formation [23]. Moreover, no information is available in open literature on the stress-reorientation behavior of the Zr–2.5 wt% Nb pressure tubes of Indian 500 MWe PHWRs, which are manufactured by a fabrication route similar to the modified route II followed by AECL [24–26]. Studies on the stress-reorientation behavior of hydrides in CWSR Zr–2.5 wt% Nb alloy, the temperature dependence of the threshold stress for reorientation of hydrides in this alloy and the influence of reoriented hydrides on the tensile properties of this alloy in the temperature range of ambient to reactor operating temperature (573 K) will generate useful data and understanding for assessing service life of the pressure tubes of PHWR. In this paper some of these issues are discussed.

2. Experimental procedure

The Zr–2.5 wt% Nb pressure tube material was obtained from Nuclear Fuel Complex, Hyderabad, in the form of sections of tubes of length 130 mm, diameter 103 mm and wall thickness 4.7 mm. 100 mm wide sections were slit from these tube-sections and cold-flattened [17]. Composition analysis showed the niobium and oxygen contents to be 2.54 and 0.1175 wt% respectively. Tensile specimens with tapered and parallel gage section were fabricated for determination of threshold stress of reorientation of hydrides and for evaluation of tensile properties, respectively. The specimen axis was along the circumferential direction of the pressure tube. After machining, both the type of specimens were stress-relieved at 673 K for 24 h. Subsequently, some of these specimens were successively polished up to 1200 grit silicon carbide abrasive paper to obtain a fresh, oxide free surface and were gaseously charged with controlled amount of hydrogen in a modified Sivert's type apparatus [11]. Both the type of specimens was charged with ~100 wt ppm of hydrogen. The hydrogen content estimated by inert gas fusion technique using a LECO analyzer is shown in parentheses in Table 1 along with the targeted hydrogen concentration.

Table 1
Details of the reorientation treatment to which the tapered specimens were subjected for determination of threshold stress for reorientation of hydrides

S. no.	Cross-sectional area (mm^2)		$T_{\mathrm{Reorientation}}$ (K)	H-content charged (estimated)	Load (kg)	Stress applied (Mpa)	
	Min.	Max.				Min.	Max.
1	11.21	31.2	623	99 (104)	200.6	63	175
2	8.4	25.2	573	99 (128)	250.6	97	292
3	9.6	28.8	523	99 (90)	270	92	276
4	9.6	28.8	473	93 (113)	308.5	105	315
5	9.89	27.3	423	93 (148)	308.5	111	306

All the specimens were solution-annealed at 673 K for 30 min before cooling at 1 K/min to reorientation temperature. Hydrogen content estimated by inert gas fusion technique using LECO analyzer is shown in parentheses.

Tapered gage tensile specimens were subjected to stress-reorientation treatment in a single arm lever type creep-testing machine under a constant applied load. For reorientation treatments at 423–623 K, the specimens were solution-annealed at 673 K for half an hour, cooled to the reorientation temperature at a cooling rate of 1 K/min, soaked at the reorientation temperature for half an hour and the load was applied for 10 min at the reorientation temperature before furnace cooling (<1 K/min) the specimens to ambient temperature. The details of the reorientation treatments are given in Table 1. For reorientation treatment at 723 K, the tapered specimens were soaked for an hour at 723 K and then furnace cooled to room temperature without any load. For evaluation of the tensile properties, the parallel gage specimens were subjected to stress-reorientation treatment consisting of solution annealing at 623 K for half an hour followed by furnace cooling under stress to ambient temperature (303 K). Tensile tests were carried out on a floor model screw driven universal testing machine at a nominal strain-rate of 3.3×10^{-4} s^{-1}. The tensile properties of the unhydrided specimens were also determined. The tensile tests were carried out in the temperature range of 298–573 K. For the purpose of temperature measurement, two number of K-type thermocouples were placed at the top and lower shoulder of the specimens. The furnace temperature was controlled within ±1 K.

For metallography, the specimens were sectioned along radial–circumferential and radial–axial plane of the pressure tube. Standard metallographic technique was followed to reveal the hydride microstructure, its morphology and distribution. For optical microscopy the specimens were etched in a solution of $HF:HNO_3:H_2O$::2:9:9 for 15 s. Threshold stress for reorientation [17] was determined by sectioning the tapered gage tensile specimens along the tensile axis. A montage was prepared for the cross-section of tapered specimens. The stress variation was computed for the montage and the stress corresponding to which half the thickness showed radial hydrides was taken as the threshold stress for reorientation [17]. Fracture surfaces were examined in scanning electron microscope.

3. Results

3.1. Hydride microstructure

Fig. 1 shows the three orthogonal directions of the pressure tube, viz., axial (A), circumferential (C) and radial (R) schematically. Crystallographically permitted orientation of the hydride platelets in Zr–2.5 wt% Nb pressure tube alloy is also shown in this figure. Typical α grain dimensions for the cold worked and stress-relieved Zr–2.5 wt% Nb pressure tube material is also indicated in this figure. The microstructural features of hydrides on two orthogonal planes (AR and RC) of the Zr–2.5 wt% Nb pressure tube material, charged with about 100 wt ppm of hydrogen, are shown in Fig. 2. This figure shows that in the as-hydrided condition, the traces of the hydride platelets (dark lines) are oriented along the circumferential direction only. These hydrides are called circumferential hydrides. This selective formation of circumferential hydrides (oriented along the axial–circumferential plane of the pressure tube) in the as-hydrided condition is attributed to the microstructure of the pressure tubes [6]. It may be noted from Fig. 2(a) and (b) that the trace on radial–axial plane is straight (with minimum irregularity or serrations) and longer than that on radial–circumferential plane. This is expected because the longer α phase grain dimension along axial direction of the pressure tube [24] provides uninterrupted growth along axial direction. The branching of the trace of the hydride on the radial–circumferential plane is due to shorter grain dimension along the circumferential direction [24] of the pressure tube (Fig. 1). Since habit plane [12] of hydrides in α phase is nearly parallel to the basal plane, for the texture of the pressure tube [25–27] one out of every three α phase grains will have favorable texture. Thus, hydrides cannot grow straight along the circumferential direction. Instead,

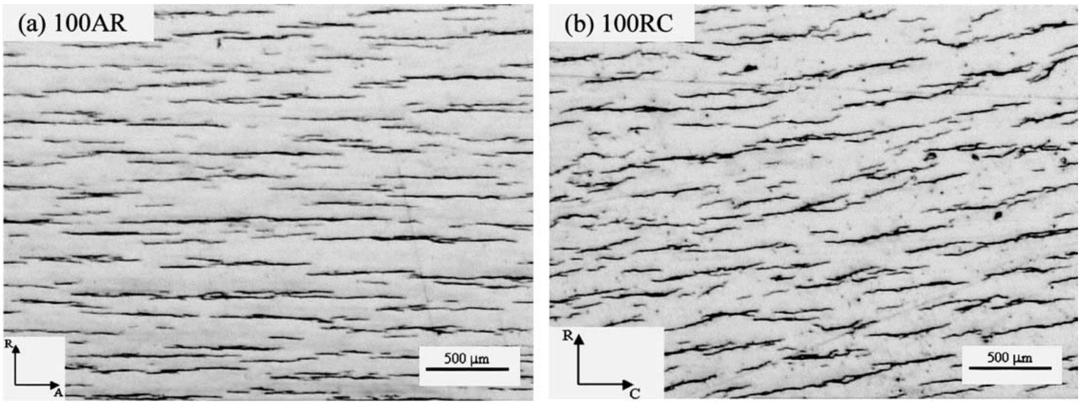

Fig. 2. The microstructural features of hydrides on two orthogonal planes (AR and RC) of Zr–2.5 wt% Nb pressure tube alloy charged with 100 wt ppm of hydrogen. The orthogonal planes are defined in Fig. 1. Arrow shows the direction. Traces of hydrides (dark lines) are longer and strainghter along axial direction as compared to that along circumferential direction.

within every 3–5 µm they will branch out to grow along the favorably oriented grains. This could be the reason for longer and straighter hydride trace along axial direction compared to shorter and branched hydride trace along the circumferential direction of the pressure tube material.

3.2. Reorientation stress, σ_{th}

For reorientation tests in the temperature range of 423–623 K, all the specimens were solution-annealed at 673 K to dissolve all the pre-existing hydrides. It is reported that the reorientation stress for Zr–2.5 wt% Nb pressure tube alloy reduces to 0 at 704 K [6,18]. Hence, for the reorientation test carried out at 723 K, the specimens were soaked for an hour and furnace cooled to room temperature without any load. Fig. 3 shows a typical micrograph for radial–circumferential section of the pressure tube, obtained by sectioning the tapered gage tensile specimens that were subjected to reorientation treatment at (a) 623 K and (b) 723 K. At 623 K, the entire cross-section of the maximum stress region (168.7 MPa) was found to consist of radial hydride (Fig. 3(a)). Towards lower stress regions, the fraction of the cross-section containing radial hydrides was noted to decrease. Similar trend was observed for the specimens subjected to reorientation treatment at 573 and 523 K. However, no significant reorientation could be observed at 423 and 473 K even at the location of the tapered gage tensile specimen that was subjected to maximum stress. For the reorientation tests carried out at 523, 573 and 623 K, the threshold stress for reorientation of hydrides in this alloy lies somewhere between the maximum and minimum stress. The stress corresponding to cross-section, where 50% of the cross-section contained radial hydrides was taken as the threshold stress [17]. Using this criteria threshold stress for reorientation could be determined at 523, 573 and 623 K. The variation in the threshold stress for reorientation of hydrides, σ_{th}, yield strength (YS) of the unhydrided material and their ratio designated as stress ratio with temperature is shown in Fig. 4. It may be noted that the value of σ_{th} shown in this figure for reorientation temperature of 473 K is conservative as significant reorientation could not be obtained at this temperature. With increase in reorientation temperature, both σ_{th} and yield stress were observed to decrease. The ratio of σ_{th} to that of YS is also found to decrease sharply from 473 to 523 K; marginally from 523 to 573 K and gradually from 573 to 623 K. For reorientation test carried out at 723 K without any load, no reorientation of hydrides (Fig. 3(b)) was observed.

3.3. Tensile properties

The temperature dependence of the tensile properties of this alloy due to reoriented hydrides is reported in terms of yield strength (YS), ultimate tensile strength (UTS), percent elongation and reduction in area (RA). Fig. 5 shows the variation in YS, UTS and percent elongation as a function of test temperature. At room temperature, both percent elongation (plastic) and reduction in area were 0, indicating the strong embrittling influence of reoriented hydrides. With the increase in test temperature, both yield and ultimate tensile strengths were found to decrease but the tensile elongation to failure and reduction in area were found to increase. Compared to the unhydrided specimens (Unhyd in Fig. 5), the YS of the specimens subjected to reorientation treatment was higher but UTS was lower, suggesting reduced work-hardening in the latter case due to the presence of hydride.

It is reported for Zircaloy-4 [28,29] that, in the as-hydrided condition even with 500 wt ppm of hydrogen, no significant loss of the ductility is observed. However,

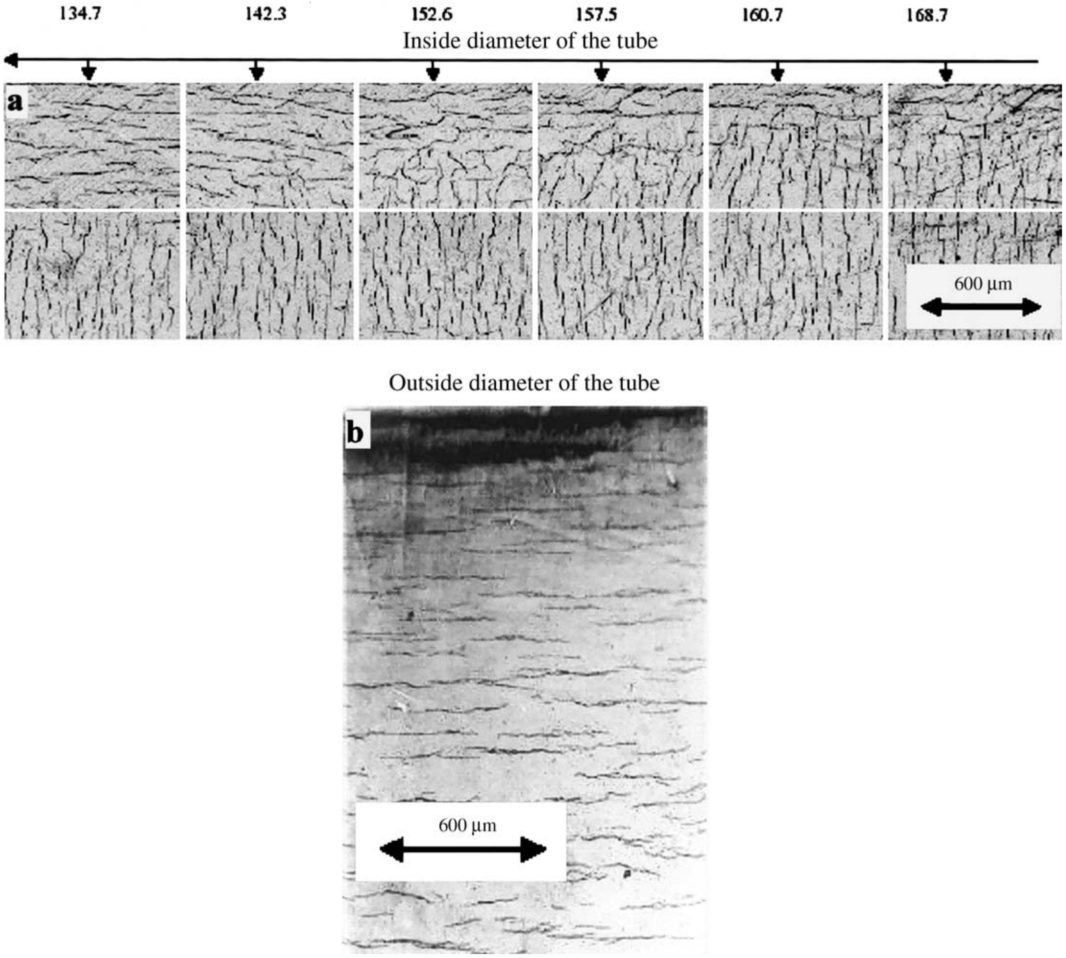

Fig. 3. The radial–circumferential section of the tapered gage tensile specimens subjected to reorientation treatment at (a) 623 K and (b) 723 K. Traces of hydrides (dark lines) can be seen. The values of the applied stresses (MPa) at different cross-section of the selected tapered specimens (a) are also shown in this figure. Towards the high stress end a higher fraction of specimen cross-section containing radial hydrides can be seen.

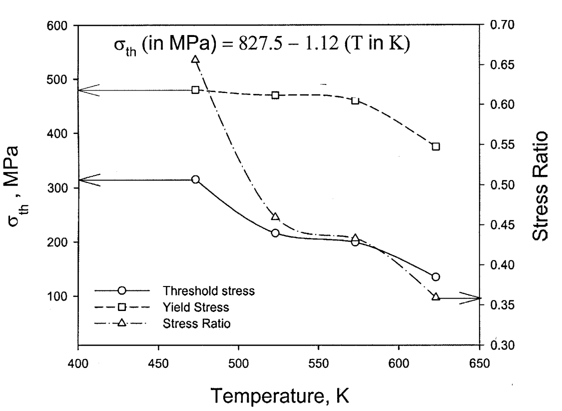

Fig. 4. Variation of threshold stress for reorientation of hydrides, yield stress and their ratio with reorientation temperature for Zr–2.5 wt% Nb pressure tube alloy.

after reorientation treatment, the ductility value as measured in terms of total tensile elongation is significantly reduced [6] even with 50 wt ppm of hydrogen. In the present work on Zr–2.5 wt% Nb alloy containing 100 wt ppm of hydrogen and subjected to reorientation treatment, the specimens tested in tension exhibited nil plastic deformation at 298 K (Fig. 5(b)). As the test temperature was increased, the total tensile elongation showed an increasing trend. However, the total tensile elongation in this alloy in unhydrided condition remains practically unchanged in the temperature range of 298–573 K [11].

The fracture surfaces of the failed specimens were examined in scanning electron microscope. Fig. 6 shows the features observed on the fracture surfaces of the specimens obtained after carrying out tensile tests at different temperatures. From these fractographs, it is

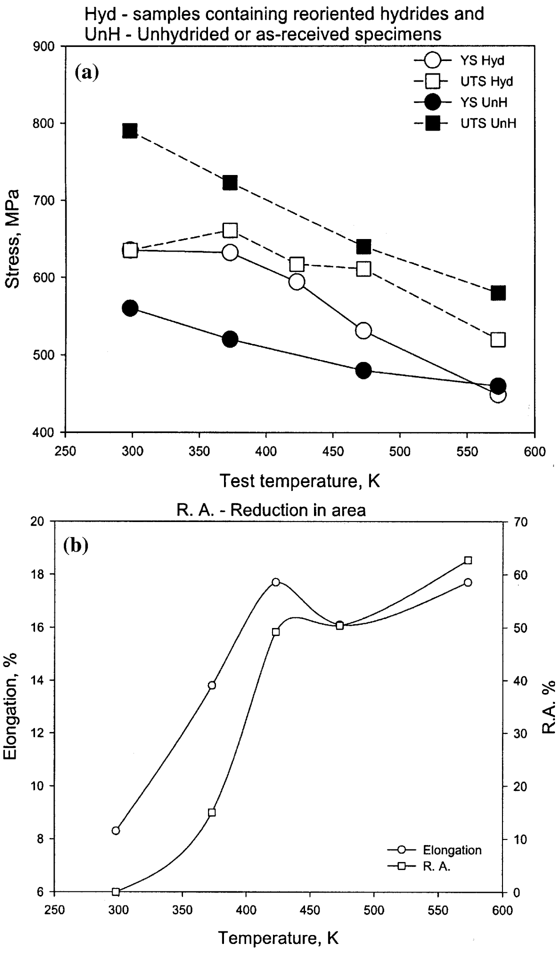

Fig. 5. Plot of (a) variation in yield stress (YS) and ultimate tensile strength (UTS), and (b) percent elongation to failure and percentage reduction in cross-sectional area as a function of tensile test temperature obtained from Zr–2.5 wt% Nb tensile specimens having reoriented hydride. The strength values are compared with that of unhydrided specimens.

evident that fracture in this alloy (containing reoriented hydrides) takes place by cleavage of hydrides and ductile tearing of the matrix (fibrous area). Below 423 K, the fractographs show little fibrous area thus indicating brittle failure. Above this temperature, the hydride volume fraction decreases due to increased solid solubility of hydrogen with increase in temperature. At the same time the matrix becomes more ductile with increase in temperature and hence the material showed a tendency to regain its unhydrided level of tensile properties. At 573 K, no trace of hydride-assisted failure is visible and the fracture mode shows fully fibrous features suggesting that the embrittlement effect is not at all significant at this temperature. Part of the hydride platelets is expected to have gone into solution at this test temperature resulting in restoration of the ductility. Thus, this increased tensile ductility with temperature is a direct result of the increasing solubility of hydrogen and the corresponding reduction in the volume fraction of brittle hydride phase. Above 423 K, this alloy containing reoriented hydrides appeared to have regained its ductility comparable to that of the unhydrided specimens (Fig. 5(b)).

4. Discussion

4.1. Variation of radial hydride fraction with stress

Fig. 3(a) and (b) shows the montage of the cross-section of the tapered specimens containing about 100 wt ppm of hydrogen and subjected to reorientation treatment at (a) 623 K and (b) 723 K. At 623 K and in the maximum stress region, the entire cross-section was seen to consist of radial hydrides only (Fig. 3(a)). Thus reorientation in this region is 100%. This is expected as the entire 100 wt ppm of hydrogen will be in the dissolved state at this temperature. As one moves away towards the lower stress region, the fraction of cross-section containing radial hydrides is found to decrease. This is because of the fact that the magnitude of the interaction energy, which is negative for radial hydride, increases with the increase in magnitude of tensile stress [30]. The magnitude of the interaction energy for the circumferential hydrides, which is positive, also increases with increase in the magnitude of tensile stress [30]. This means that, with increase in the magnitude of applied tensile stress, the probability of the radial hydride precipitation increases at the expense of circumferential hydride. At reorientation temperature lower than 623 K, hydrogen will be partly in solution and will partly exist as circumferential hydrides, depending on the TSS at reorientation temperature under consideration. The precipitation of dissolved hydrogen as radial hydrides during the reorientation process will face competition from the growth of the existing circumferential hydrides and thereby influencing the degree of reorientation. As the number of the circumferential hydride platelet increases its influence on the degree of reorientation is likely to increase. For the reorientation test at 573 K, about 60 wt ppm of hydrogen will be in dissolved state whereas remaining hydrogen will exist as circumferential hydrides. Even though the entire cross-section of the specimen, which experienced maximum stress, contained radial hydrides, there existed circumferential hydrides as well. At 523 K, about 30 wt ppm of hydrogen will be in solution and the remaining hydrogen will be present as circumferential hydrides. Thus, as the reorientation temperature decreased the amount of hydrogen present in solution and thereby participating in the reorientation process decreased. This was reflected by the decrease in the length of the radial hydride trace and by the increase

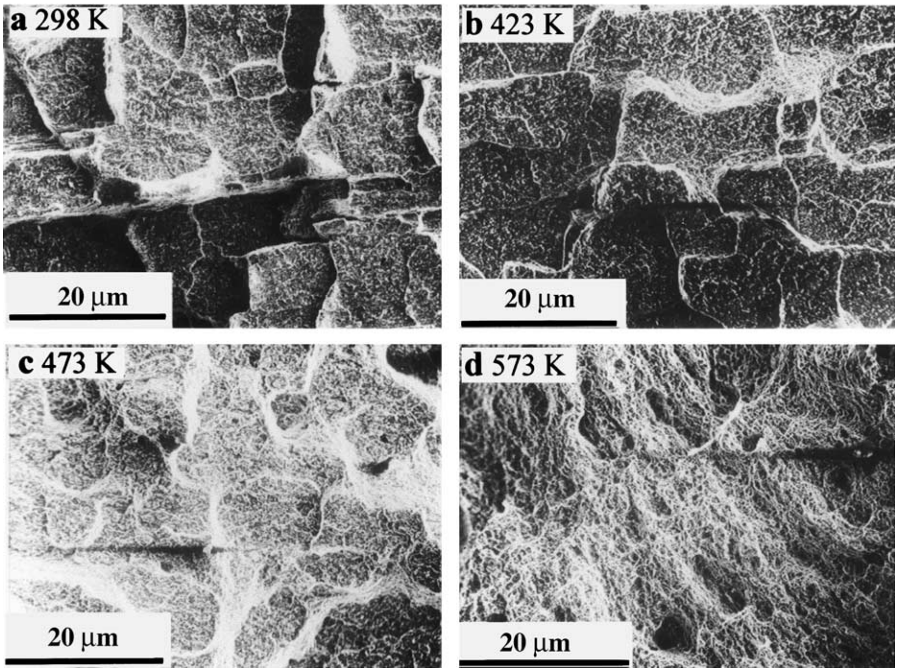

Fig. 6. Features observed in the fracture surfaces of the specimens obtained after carrying out tensile tests at temperatures of (a) 298 K, (b) 423 K, (c) 473 K and (d) 573 K. Radial hydrides were oriented normal to stress axis. At the test temperature of (a) 298 K and (b) 423 K the fracture surfaces are predominantly of cleavage type. As the test temperature is increased, the partial dissolution of hydrides and the increase in matrix ductility result in mixed cleavage and fibrous regions at 473 K (c). At 573 K (d), the fractograph exhibits predominantly ductile fracture (fibrous regions) behavior.

in the inter-platelet spacing of the radial hydrides with decrease in the reorientation temperature. For the specimens reoriented at 423 and 473 K, the amount of hydrogen in solution will be 7.5 and 15 wt ppm respectively. As a result of this, no significant reorientation was observed even in the cross-section of the tapered gage tensile specimen experiencing the maximum applied stress. Therefore, this leads to suggestion that either the applied maximum stress was lower than the threshold stress for reorientation of hydrides in this alloy at these temperatures or the traces of radial hydrides were so small that they could not be resolved under optical microscope.

4.2. Temperature dependence of the σ_{th}

The threshold stress for reorientation of hydrides depends on the yield strength of the material [6]. With a decrease in the yield strength of the material, σ_{th} is reported to decrease. For a given alloy, as the reorientation temperature is increased, the yield strength decreases and hence σ_{th} is also expected to decrease with increase in reorientation temperature. In the present investigation also, σ_{th} is found to decrease with the increase in reorientation temperature. In one of the earliest work on experimental determination of σ_{th} carried out by Bell and Duncan [18] in mid-1970s, it was suggested that the value of σ_{th} should reduce to 0 at a temperature of 704 K. That is why one of the tapered specimens was subjected to reorientation treatment at 723 K without any load (Fig. 3(b)). However, no reorientation was observed at this temperature. This, therefore, suggests that, for the cold worked and stress-relieved Zr–2.5 wt% Nb pressure tube material used in this investigation even though the value of σ_{th} decreases with increase in reorientation temperature, at least up to a temperature of 723 K it does not reduce to 0. This difference in the stress-reorientation behavior of hydrides observed at 723 K in CWSR Zr–2.5 wt% Nb pressure tube alloy between the present investigation and those reported earlier [18] could be due to different microstructural conditions of the materials used in the two studies and the non-linear temperature dependence of the threshold stress. A linear regression analysis of the plot between σ_{th} and temperature (Fig. 4) yielded the relationship for temperature dependence of σ_{th} as given in Eq. (1) below, with $r^2 = 0.93$.

$$\sigma_{\mathrm{th}}\ (\mathrm{MPa}) = 827.5 - 1.12T\ (\mathrm{K}). \quad (1)$$

However, a non-linear regression analysis suggested a better cubic polynomial fit and yielded the relationships for the temperature dependence of yield stress (YS), threshold stress (σ_{th}) and normalized threshold ($\sigma_{\text{th}}^{\text{Normalised}}$) of the forms given in Eqs. (2)–(4).

$$\text{YS (MPa)} = 1.475 \times 10^4 - 82T + 0.16T^2 - 10^{-4}T^3, \tag{2}$$

$$\sigma_{\text{th}} = 2.99 \times 10^4 - 160T + 0.29T^2 - 1.732T^3, \tag{3}$$

$$\sigma_{\text{th}}^{\text{Normalised}} = 52 - 0.275T + 4.9 \times 10^{-4}T^2 - 2.9 \times 10^{-7}T^3, \tag{4}$$

where YS and σ_{th} are in MPa and T is in K.

5. Conclusions

The threshold stress (σ_{th}) for reorientation of hydrides was determined for the double melted, cold worked and stress-relieved, unirradiated Zr–2.5 wt% Nb pressure tube alloy as a function of reorientation temperature. The magnitude of σ_{th} was found to decrease non-linearly with increase in reorientation temperature. For a given temperature and hydrogen content the degree of reorientation was found to increase with reorientation stress. At temperatures lower than 473 K, competition from existing circumferential hydrides reduces the efficiency of reorientation. The embrittlement effect of the reoriented hydrides was most deleterious below a temperature of 423 K with the specimens showing nil plastic deformation at ambient temperature.

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