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Effect of cold working and annealing on the texture of Zr-2·5Nb pressure tubes

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Abstract. Texture plays an important role in the commercial acceptability of Zr-2·5 wt% Nb pressure tubes used in nuclear reactors. A modified flow sheet for the fabrication of these pressure tubes involves a few additional steps viz. stress relieving, cold working and annealing, as compared to the conventional route. The evolution of texture during sequential fabrication steps of extrusion, stress relieving, cold working and annealing was studied in terms of texture coefficients and inverse pole figures. It was observed that crystallographic texture primarily developed during hot extrusion and the additional steps of stress relieving, cold working and annealing did not alter the texture significantly. The texture developed was one having a majority of the basal plane normals along the tangential direction of the tube.

Keywords. Annealing; cold working; inverse pole figures; pressure tubes; texture; zirconium base alloys; Zr-2·5 Nb alloy.

1. Introduction

The Zr-2·5wt% Nb alloy is used as a pressure tube material in pressurized heavy water reactors (PHWRs). The microstructure of the tube comprises elongated α grains surrounded by β-phase stringers (Cheadle *et al* 1982). Alpha zirconium has a hexagonal crystal structure and hence a limited number of deformation modes. Therefore during fabrication of the tubes a strong crystallographic texture is produced (Picklesimer 1966; Cheadle *et al* 1967). Since the irradiation creep and growth, the hydride orientation and the mechanical properties of the pressure tubes are all texture-dependent, it is imperative to obtain tubes with favourable textures.

A new fabrication route of these tubes involves a few additional stages during fabrication, viz. stress relieving, cold working and annealing, as compared to the conventional fabrication practice (figure 1) (Asundi and Banerjee 1989; Sundaram *et al* 1989). The duplex microstructure of the alloy, where both α and β phases are deformable, is amenable to recovery and recrystallization processes during annealing. This investigation deals with the development of texture in hot extruded Zr-2·5 wt% Nb pressure tubes on stress relieving, cold working and annealing. The texture has been characterized in terms of texture coefficients and inverse pole figures.

2. Experimental

The starting material was received from the Nuclear Fuel Complex, Hyderabad in the form of hot extruded (in $\alpha + \beta$ phase field) Zr-2·5 wt% Nb tubes. Rectangular strips were machined along the longitudinal direction of the tube while maintaining the strip surface normal to the radial direction of the tube. The strips were encapsulated in pyrex tubes under vacuum and stress-relieved at 753 K for 24 h. An amount of deformation equivalent to that used during the first pass pilgering of pressure tubes for

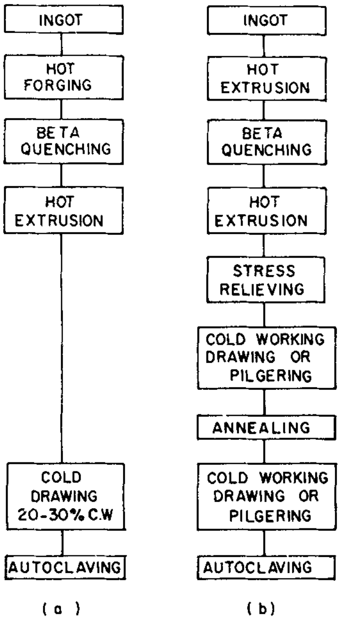

Figure 1. Comparison of (**a**) conventional and (**b**) modified flowsheets for the fabrication of Zr-2·5 Nb pressure tubes.

235 MW PHWRs, viz. 55% reduction in thickness, was imparted to the strips by rolling them at room temperature. The cold-rolled strips were then encapsulated in silica tubes under Argon atmosphere and annealed at 798 K for 9 h. The time and temperature of annealing were so chosen as to remove the cold work introduced during rolling, without affecting any other microstructural feature (Haq *et al* 1989).

Samples were taken from different stages of fabrication for microstructural examination and texture evaluation. Radial, longitudinal and tangential sections of these samples were prepared from the strips. The strip surface represented the tangential section of the tube. Composite samples representing radial and longitudinal sections of the tube were obtained by slicing the strip parallelly into small pieces along its width and length respectively and mounting adequate number of pieces together to provide sufficient surface area for X-ray diffraction. The worked layer was removed by chemical polishing in a solution containing 45% HNO_3, 10% HF and 45% H_2O.

3. Texture analysis

Crystallographic textures were determined by X-ray diffraction using the inverse pole figure technique (Harris 1952). In this technique, for a material of given composition, the total diffracted intensity was taken to be independent of the texture present in the material. The diffraction intensities from samples representative of the three principle directions of the material were compared with those of a sample having random orientation. These relative intensities were expressed as texture coefficients T_i, which were defined by the following equation:

$$T_i = (I_i/I_i^r) \Big/ \left[(1/n) \sum_{i=1}^{n} (I_i/I_i^r) \right],$$

where I_i and I_i^r are the integrated intensities of the reflection from the ith crystallographic plane of the textured sample and a reference random sample respectively and n the total number of reflections measured. The texture coefficient of a plane *hkl* is proportional to the frequency of grains having *hkl* planes parallel to the surface examined. A texture coefficient of unity denotes random orientation i.e. larger the value more intense the texture. Inverse pole figures, which show the distribution of all the poles in a given direction, were obtained by plotting the calculated texture coefficient values on a stereographic projection. The direction of the basal plane normal being important for relating the texture to various material properties, textures were compared in terms of several idealized orientations of the grains. The idealized α-grain orientations and their relationship to inverse pole figures are shown schematically in figure 2. The A, C and D orientations denote the grains having their basal plane normals parallel to the tangential, radial or longitudinal directions respectively and their texture coefficients are those of the basal planes. Orientations AB, CB and DB refer to the grains with their basal poles within 40° of the A, B and D orientations respectively and their texture coefficients are the average of the $(10\bar{1}5)$, $(10\bar{1}4)$, $10\bar{1}3)$ and $(11\bar{2}4)$ planes.

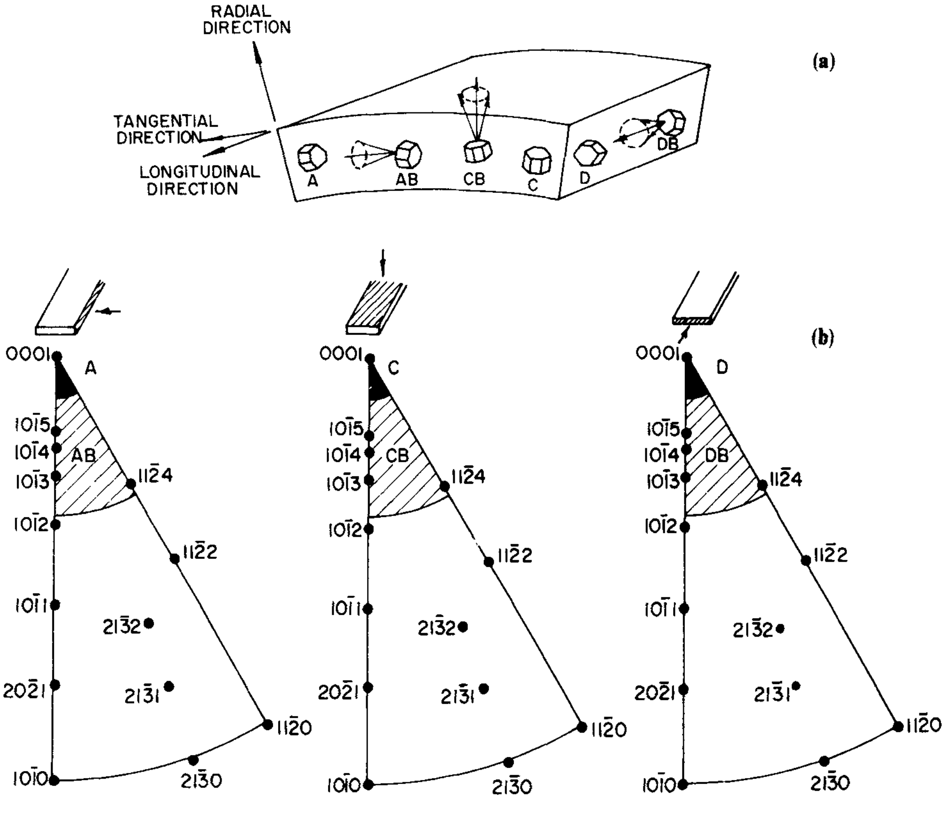

Figure 2. Idealized orientations on (a) a section of the tube and (b) inverse pole figures. The reference directions are as indicated.

4. Results and discussion

The microstructure of Zr-2·5 Nb alloy at the extrusion temperature consists of about 20% α-phase and 80% cubic β-phase (Cheadle *et al* 1982). Extrusion leads to elongation of the α-grains. During subsequent air cooling, the β-phase partially transforms to the α-phase by growth on the existing α-phase. Hence the microstructure of the extruded tube consists of elongated α grains and a grain boundary network of β-phase containing about 18–20 wt% Nb. This duplex microstructure is revealed in the optical micrograph of the extruded tube (figure 3a). As expected, stress relieving affected neither the morphology or distribution of the two phases nor the aspect ratio of the α grains. It only leads to a decrease in dislocation density. Cold working resulted in further elongation of the grains (figure 3b). This also increases the dislocation density. No recrystallization was found to occur on annealing at 798 K for 9 h and the microstructure resembled that of the cold-worked microstructure. It was earlier found that this anneal completely removes the cold work arising from the preceding stage, through a decrease in dislocation density, without affecting other microstructural features (Haq *et al* 1989).

The extrusion process plays an important role in determining the texture of the finished tubes. Factors which can affect the texture and microstructure of the extruded tubes are composition of the alloy, billet microstructure, temperature during extrusion, deformation mechanisms operative in the α grains during extrusion, die design, lubrication and cooling rate after extrusion (Cheadle *et al* 1972). However, when the tubes are extruded in the $\alpha + \beta$ phase field the temperature and hence the amount of α present have little effect on the texture developed. Cheadle *et al* (1967) have shown that when zirconium alloys are deformed in the α or $\alpha + \beta$ regions the α grains are oriented so that their basal plane normals are close to the direction of major compressive strain. Since there is considerable reduction in thickness during extrusion, it may intuitively appear that the major compressive strain would be in the radial direction of the tube. However, due to the non-uniform flow of metal in extrusion, the major strain and hence the basal plane normals of α grains lie close to the circumferential direction (Cheadle *et al* 1972). In other words, a majority of the grains in the extruded tubes are expected to have A/AB orientations. The texture coefficient values of the various *hkl* planes are given in table 1 and those for the idealized α grain orientations in table 2. It can thus be seen from table 2 that the as-received material has strong texture with grains in the A/AB and C/CB orientations, the majority of the grains being in the A/AB orientations. Similar results have also been reported earlier by other workers (Cheadle and Evans 1966; Cheadle *et al* 1967).

Figure 4 shows the inverse pole figures of the tangential, radial and longitudinal directions for the material after different stages of fabrication (a to d). The numbers within parenthesis on the inverse pole figures are the texture coefficients of the respective reflections and the curves within the inverse pole figures define the regions having the indicated range of texture coefficient values. It can be seen from figure 4 that the basal poles at all the stages of fabrication are concentrated to a very high degree on the tangential direction whereas the $\{10\bar{1}0\}$ prism plane poles are mainly concentrated along the longitudinal direction.

Table 2 and figure 4 indicate a certain change in texture on stress relieving, cold working and annealing. For example, cold working as well as annealing decreased the number of grains in the A/AB orientations (table 2). The variations in texture involved

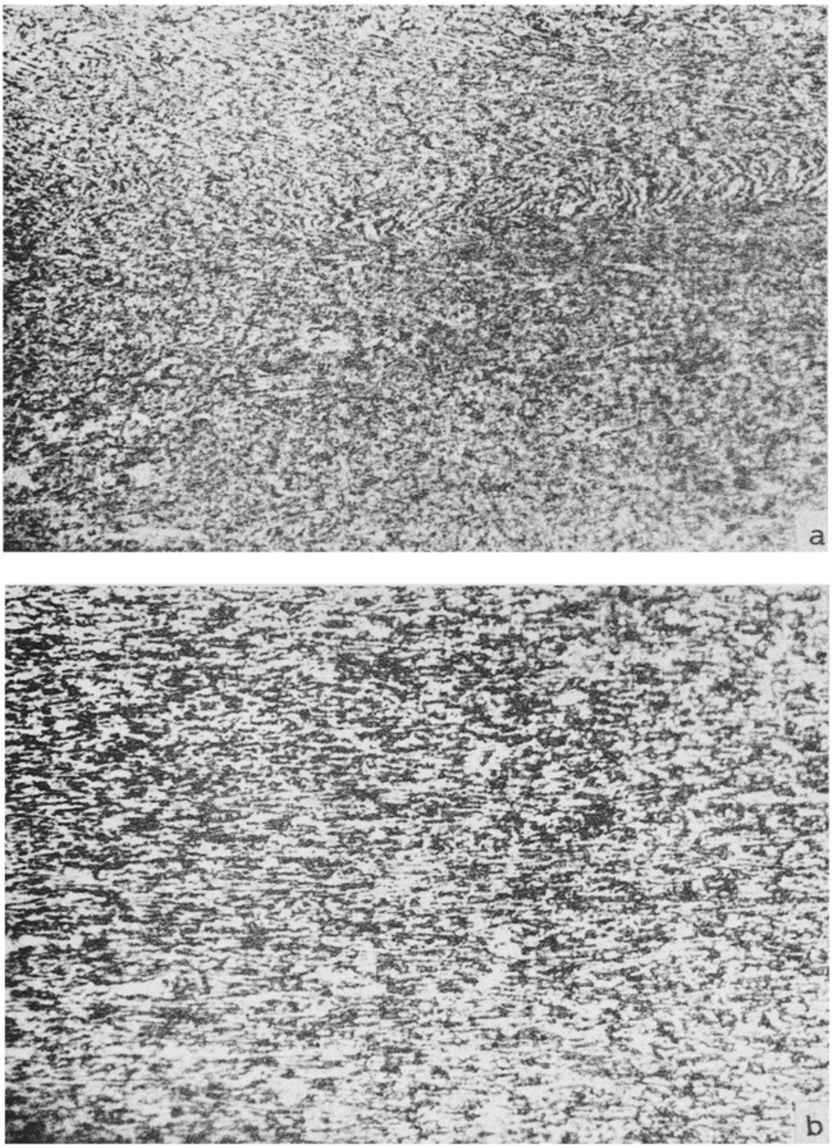

Figure 3. Microstructures of Zr-2·5 Nb pressure tube in the longitudinal section showing elongated α grains (white) surrounded by a network of β phase (dark). (**a**) As received and (**b**) 55% cold worked 200 ×.

are, however, too small to be of any practical significance and the overall texture basically remains the same. The results therefore show that crystallographic texture is primarily developed during hot extrusion of Zr-2·5 Nb tubes and it remains practically unaltered during subsequent fabrication steps. It may be mentioned here that the possibility of an appreciable change in texture cannot, however, be ruled out if the tubes are heated above 873 K at any stage after hot extrusion (Cheadle *et al* 1972).

Pressure tubes produced by the conventional route have performed well in the

Table 1. Texture coefficients along the tangential (*T*), radial (*R*) and longitudinal (*L*) directions of the Zr-2·5 Nb tube after various fabrication stages.

	Hot extruded			Stress relieved			Cold worked			Annealed		
hkl	*T*	*R*	*L*	*T*	*R*	*L*	*T*	*R*	*L*	*T*	*R*	*L*
100	0·6	0·2	5·8	0·3	0·2	5·4	0·7	0·2	8·3	0·5	0	5·5
002	5·8	1·2	0	5·6	1·0	0·6	4·8	1·1	0·2	4·5	0·4	0
101	0·6	0·2	0·9	1·1	0·2	0·7	0·8	0·2	0·7	0·7	0·1	0·3
102	0·8	0·7	0	1·5	0·4	0	0·6	0·6	0	0·7	0·3	0
110	1·1	1·7	0	0·7	2·9	0·2	0·9	2·8	0·7	0·7	0·4	1·6
103	1·3	1·3	0	1·5	1·4	0	0·6	1·3	0	0·7	0·7	0
200	0	0	7·0	0	0	7·9	0	0	5·9	0	0·5	7·0
112	0·8	1·5	0·5	0·8	2·3	0·3	1·0	2·3	0	0·9	0·4	0·8
201	0·8	0	2·8	0·6	0	2·8	1·0	0	1·2	0·6	0·4	1·8
004	4·8	0·9	0	2·8	1·2	0	3·6	0	0	4·8	1·2	0
202	0	0	0	0	0	0	0	0	0	0	1·2	0
104	0	1·0	0	0	2·0	0	0	1·6	0	0	0·8	0
203	0	1·1	0	0	0	0	0	0	0	0·6	1·7	0
210	0	0	0	0	0	0	0	0	0	0	0·7	0
211	0·6	1·7	1·0	0·9	1·0	0	1·0	0·6	1·0	0·8	1·6	1·0
114	1·3	3·0	0	2·3	4·0	0	3·0	4·3	0	2·4	3·0	0
212	0	1·4	0	0	0	0	0	0	0	0	2·5	0
105	0	2·0	0	0	1·3	0	0	3·0	0	0	2·0	0

Table 2. Texture coefficients of the idealized orientations.

	Texture coefficients					
Sample condition	*A*	*AB*	*C*	*CB*	*D*	*DB*
Hot extruded	5·8	0·6	1·2	1·8	0·0	0·0
Hot extruded and stress relieved	5·6	1·0	1·0	2·2	0·6	0·0
Hot extruded, stress relieved and 55% cold worked	4·8	0·9	1·1	2·6	0·2	0·0
Hot extruded, stress relieved, 55% CW and annealed	4·5	0·8	0·4	1·7	0·0	0·0

currently operating PHWRs but the new route has the advantage that it allows one to achieve a better dimensional and microstructural control. The present results show that, by optimizing the fabrication process parameters, it is possible to obtain similar crystallographic textures in the pressure tubes produced by the two routes. Thus the apprehension that the tubes fabricated in the modified flow sheet might develop a different texture is unfounded. However, if a stronger basal pole texture along the tangential direction, as has sometimes been reported (Cheadle and Evans 1966; Cheadle *et al* 1967), is desired, a higher extrusion ratio could be employed. Holt and Aldridge (1985) have shown that increasing the extrusion ratio decreases the ratio of the number of basal plane normals in the radial direction to the number in the tangential direction.

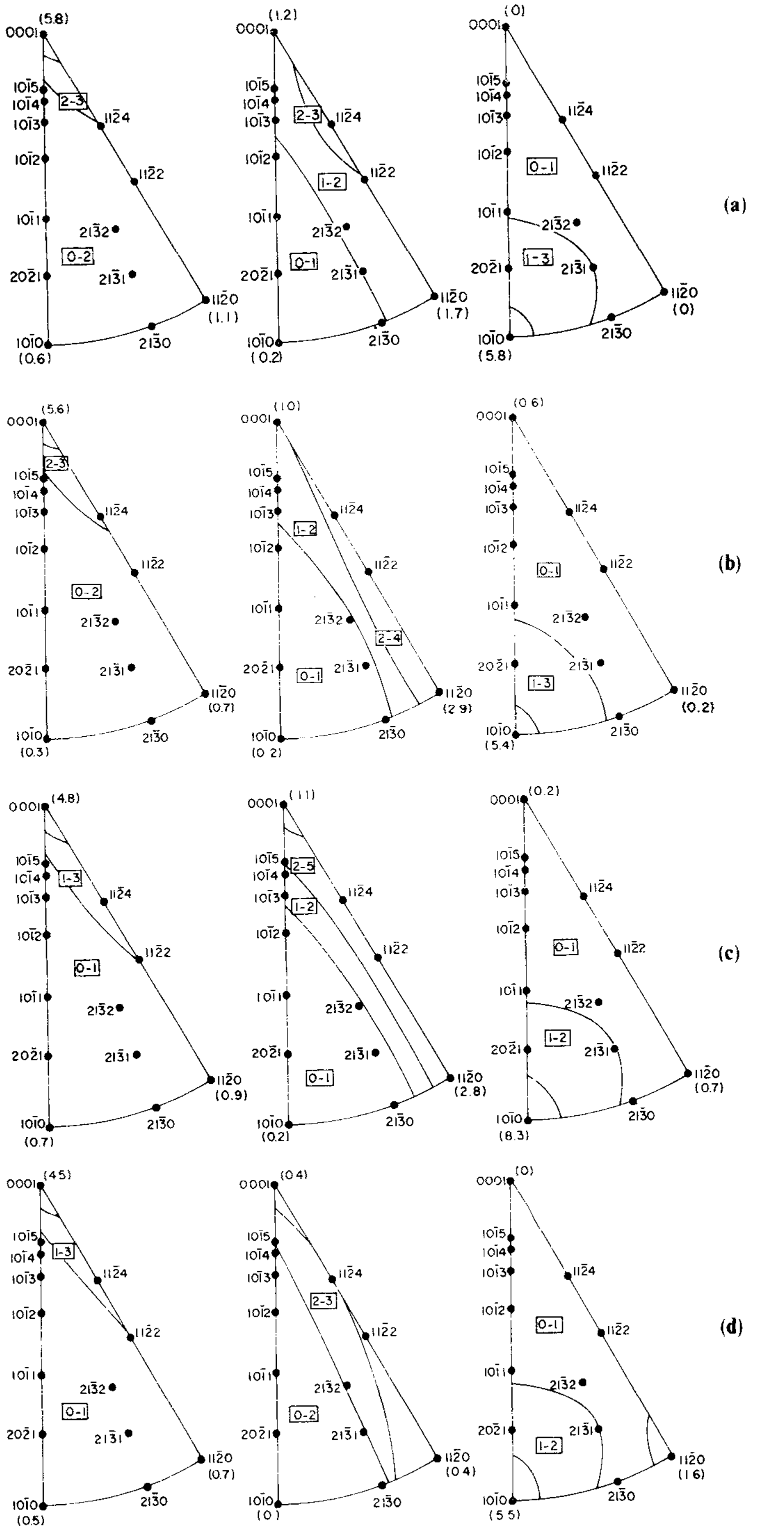

Figure 4. Inverse pole figures of Zr-2·5 Nb tube subjected to a fabrication sequence involving (**a**) hot extrusion in $\alpha + \beta$ phase field, (**b**) stress relieving at 753 K for 24 h, (**c**) cold working to 55% deformation and (**d**) annealing at 798 K for 9 h. The reference directions are the same as in figure 3.

5. Conclusion

The hot extruded Zr-2·5 Nb pressure tubes possess strong texture with a majority of the basal poles oriented along the tangential direction. The additional fabrication steps of stress relieving, cold working and annealing, in the modified flow sheet, do not alter the texture of the tubes significantly.

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