

## CHARACTERIZATION OF THE CRYSTALLINE TEXTURE OF TUBES MADE OF ZIRCALOY-4.

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### ABSTRACT

In view of the critical character of the conditions of use of Zircaloy-4 cladding tubes in the nuclear industry, it is necessary to characterize them and try to inspect them as best as possible, especially insofar as concerns the mechanical and structural characteristics as crystalline texture.

We studied, for each fabrication step, the crystalline texture of the material through incomplete pole figures (00.2). We observed a clear evolution from tangential tendency, for the initial product, to radial tendency of texture for the final product. This is in concordance with mechanical reduction coefficients used for pilgering.

In the same way, a texture gradient is visible through the wall; this is probably due to the rolling process which leads to strain anisotropy.

### INTRODUCTION

The anisotropic nature of the macroscopic properties of a material can depend on the crystallographic anisotropy and the crystalline texture. Especially, for Zircaloy tubes, it has been demonstrated that a radial orientation of the crystallites is more favorable than a tangential orientation versus inservice general behaviour (1). Moreover, there is an important correlation between the deformation mechanisms in the tube and the texture (2). We have thus undertaken the study of the crystalline texture for various stages in the cladding tubes manufacturing process.

The process implies the total reduction of the initial section by more than 98% and of the thickness by more than 92%. These reductions are obtained by three successive cold pilgering passes intersected by recrystallization heat treatment.

## EXPERIMENTAL

The tubes investigated are representative of each fabrication step, that means each pilgering pass as rolled and heat treated conditions (recrystallization following the first and second passes and stress-relieving following final pass). For each tube, three samples corresponding to three locations through the wall (o. d., mid., i.d.) were obtained by etching. Thus, in view of characterization of the texture of the fabrication process, no less than 21 samples were prepared and analysed.

For this analysis, the wall thickness of each sample was reduced down to less than 100  $\mu\text{m}$ . and the latter was then unrolled and stucked on a sample-holder made with plastic directly fitted on the goniometer.

The crystalline texture was analysed with the help of direct pole figures produced by X ray diffraction.  $K\alpha$  radiation of copper anticathode was used. The goniometer fitting up, for incomplete pole figures production, was designed by L.A.M.M. using MICRO CONTROLE components.

The diffracted intensity was collected by a position sensitive detector (P.S.D.) INEL with a total opening angle of about  $11^\circ$  ( $2\theta$ ), this allows the record, in the case of Zircaloy tubing, and with the radiation used, of the signal diffracted by planes (10.0), (00.2) and (10.1) simultaneously. The record of counting results was carried out with a DEC LST 11/02 computer and data handling was carried out with an IBM PS/02 computer.

The goniometer is computer-directed so that the declination angle is given a forward movement of  $5^\circ$  (between  $0$  and  $75^\circ$ ) for each incessant rotation (from  $0$  to  $360^\circ$ ) of azimuthal angle. Besides, the sample is actuated by a translation movement in its plane with an amplitude of  $\pm 4\text{mm}$ . The data recording is made in each interval of  $5^\circ$

(azimuth) with acquisition time of 10 seconds. All measurements were carried out with a back reflection configuration using the Schulz method (3).

The background noise was withdrawn. The correction of defocusing was proved needless by reason of the goniometric fitting and the use of a P.S.D. The normalization of intensities was calculated with regard to the portion of sphere at  $75^\circ$ .

As an illustration, we present seven incomplete pole figures corresponding to the midwall location at each pilgering and heat treatment step (fig. 1).

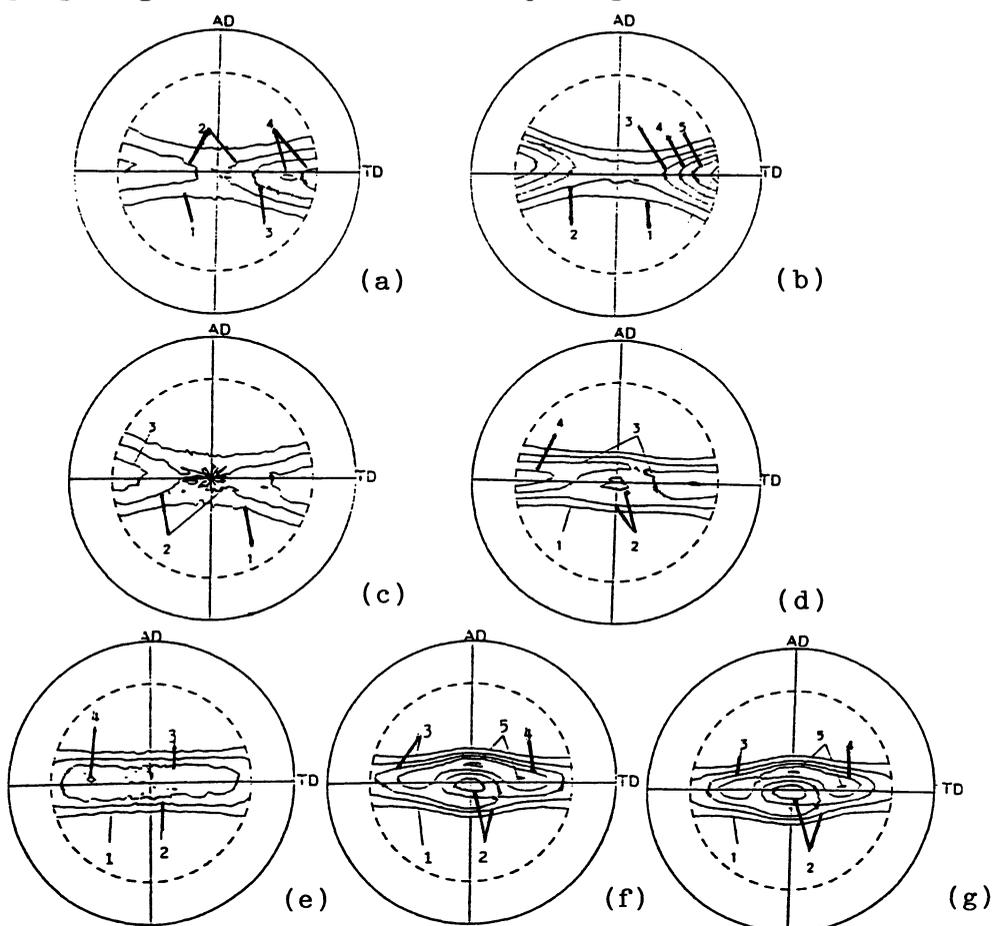


Figure 1. Incomplete pole figures (midwall):  
 a) Tube reduced extrusion (TRES) recrystallized,  
 b) First pass as rolled, c) First pass recrystallized,  
 d) Second pass as rolled, e) Second pass recrystallized,  
 f) Third pass as rolled, g) Third pass stress relieved.

## DISCUSSION

For each manufacturing stage, we have produced three incomplete pole figures of the planes (00.2) by X-ray diffraction corresponding to three locations in the thickness of the tube. All the pole figures show a bimodal distribution of intensities in the plane RD-TD (RD:radial direction, TD:tangential direction). The Zircaloy 4 has an hexagonal close packed structure with a c/a ratio  $<1.633$ . As it was demonstrated (4), this ratio, in connection with the deformation way followed by the tube, leads to the bimodal texture above pointed out. For the quantitative evaluation of the texture, we used:

a)  $\gamma$  angle between radial direction and the direction corresponding to the maximum intensity (the latter is generally located in the RD-TD plane). If  $\gamma < 45^\circ$ , basal poles are inclined to radial direction. If  $\gamma > 45^\circ$ , basal poles are inclined to tangential direction.

b) Kearns factor in radial direction  $f_R$  (5)

In pilgering process,  $Q_p$  factor is defined, which represents the wall thickness to diameter reduction ratio. This factor may be, according to Hobson et al.(2) used for predicting the tendency of basal poles towards radial direction if  $Q_p > 1$ .

The global texture has been characterized and we have observed a clear evolution from a texture with a tangential trend for the initial product (from a hot extruded and reduced tube - Kearns  $f_R$  approx. 0.45) towards a texture with a radial trend after the third cold rolling pass (Kearns  $f_R$  approx. 0.6) (see table 1). On the other hand, a

Table 1. Radial Kearns factors (mid wall) and  $Q$  parameter for each pilgering pass.

	TRES	FIRST PASS		2nd PASS		3rd PASS	
		as rolled	Recryst	as rolled	Recryst	as rolled	stress relieved
$f_R$	0,47	0,43	0,46	0,53	0,57	0,60	0,60
$Q_p$		1,17		1,47		1,48	

variation of  $\gamma$  angle is also pointed out from 75° for TREX to about 40° for the cladding tube this in concordance with Moulin et al. (6) (see fig.2). These evolutions can be explained by the relationship between the relative reduction in thickness to the relative reduction in diameter used during the different rolling passes. The fact of obtaining  $Q_p > 1$  in each pilgering pass is not yet enough for predicting without any ambiguity a radial tendency for texture, because the latter exists only after the third pass where  $\gamma$  changes from 50 to about 40°. Consequently, it seems that the initial texture must be taken into consideration in view of texture prediction.

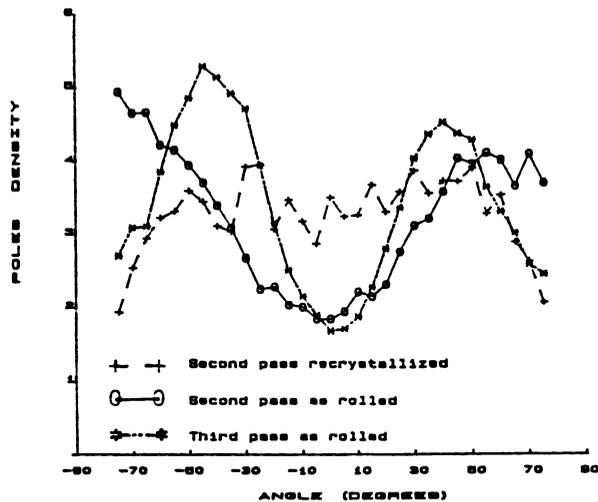


Figure 2. Evolution of  $\gamma$  angle with the pilgering passes

After the recrystallisation heat treatment, we observe on the pole figures a trend towards the disappearance of the bimodal disposition of the intensity maxima of the RD-TD plane. This phenomenon is probably due to a redistribution of the cristallites in the RD-TD plane. In fact, during the growth, the new grains tend to have a more radial orientation firstly causing the closing up of the maxima towards the center of the poles figures and secondly, a lowering of their intensity. Conversely, stress relief heat treatment, applied in the final manufacturing phase, would not seem to affect the cristalline texture of the tube whereas it does have an influence on the CSR (Contractile Strain Ratio), which is however considered to be directly correlated thereto (7).

It would seem that the texture is more marked on the internal surface of the tube than on the external one (see fig.3). This phenomenon is all the more striking when the tube is thicker. This evolution in the thickness is probably linked to the method of reducing which induces strain anisotropy.

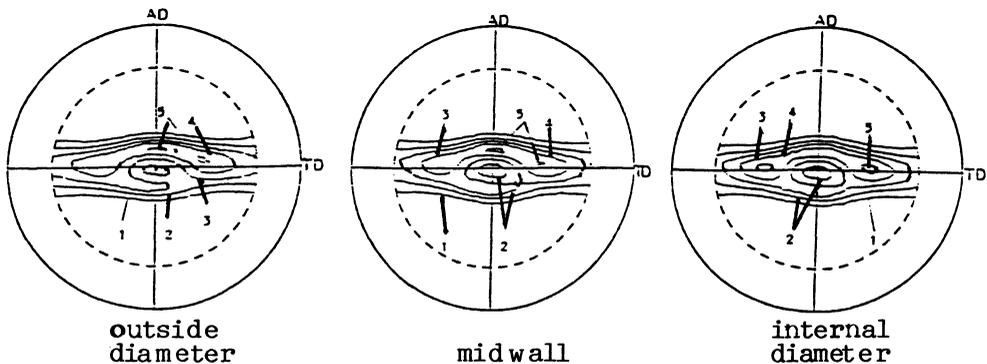


Figure 3. Texture gradient through wall thickness in final dimension (as rolled).

Further studies are in process firstly to better characterize the texture conditions through the ODF (Orientations Distribution Function) and secondly, to characterize the residual strain condition by macroscopic measurements and XR diffraction. The relationship between these measurements could provide for better optimization of the reducing and heat treatment conditions thus obtaining an end product meeting high technology requirements.

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