

## Texture and Microstructure Development of Zr Alloy during Cold-rolling

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### 1. Introduction

The texture of zirconium alloy is well known to be influenced by processing; thus the control of texture in the final product for nuclear fuel applications is a major concern [1-5]. In practice, however, the knowledge has not been sufficient in obtaining reasonable control and enhancement of texture in zirconium alloys. Studies were therefore undertaken to more closely assess the effects of processing on texture in zirconium base alloys. The objective of this effort is to identify those factors important to texture control and enhancement which could be practically applied to the manufacture of zirconium cladding. The work reported here is a step in that direction.

### 2. Methods and Results

#### 2.1 Material and processing

The material used in the present study is a Zr-1.0wt.%Nb sheet hot-rolled by 71% at 580°C.

Fig.1 illustrates the detailed manufacturing process of the sheet in this experiment.

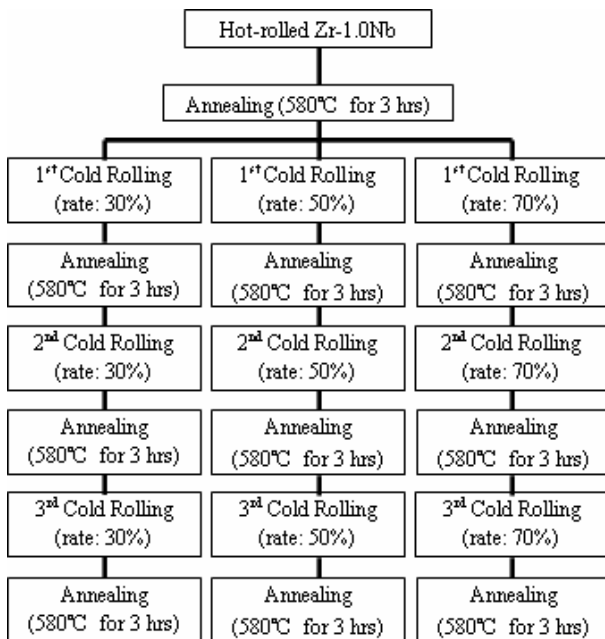


Fig. 1. The manufacturing process flow sheet of Zr-1.0Nb alloy used in the present study.

#### 2.2 Measurements

Bulk texture measurement data were obtained by measuring five incomplete pole figures of the following poles:  $\{0001\}$ ,  $\{11\bar{2}0\}$ ,  $\{10\bar{1}1\}$ ,  $\{10\bar{1}3\}$  and  $\{10\bar{1}2\}$ . These were selected specifically to avoid, to the best possible extent, overlapping of any neighbouring X-ray peaks or poles coming from the hcp phase. X-ray ODFs were calculated by inversion of the five incomplete pole figures and using the standard series expansion.

Optical microscopy samples were mechanically polished and chemically etched using a solution of 5% hydrofluoric acid and 45% nitric acid, rest being distilled water. The samples for TEM observation were prepared by thinning the alloy tube to a thickness less than 70  $\mu\text{m}$  by using a solution of 10 vol.% HF, 30 vol.%  $\text{H}_2\text{SO}_4$ , 30 vol.%  $\text{HNO}_3$  and 30 vol.%  $\text{H}_2\text{O}$  followed by a twin jet polishing with a solution of 10 vol.%  $\text{HClO}_4$  and 90 vol.%  $\text{C}_2\text{H}_5\text{OH}$ .

#### 2.3 Microstructure Development during Cold-rolling

Fig.2 shows the variation of microstructure during different cold-rolling rates. As can be seen from the figure clearly, grain size decreased with an increase of cold-rolling rates range from 30% to 70%, and under same cold-rolling rate, the grain size was nearly invariant.

The microstructure of the alloy cold-rolled at the three rates consisted of recrystallized grains with a number of precipitates distributed both in the grains and on the boundaries.

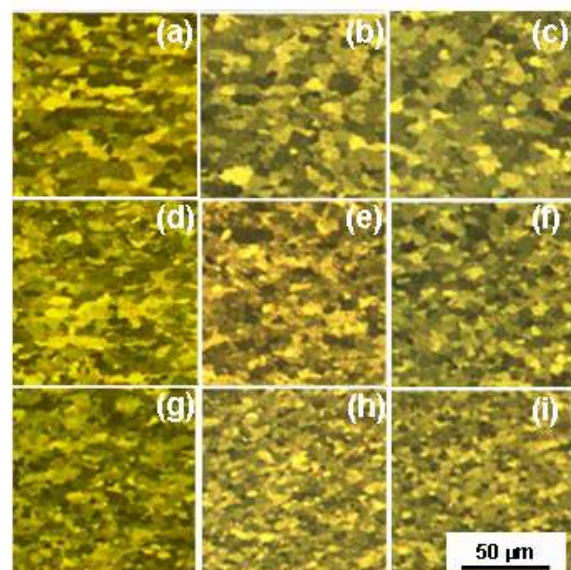


Fig. 2. Optical micrograph for 30%CR (a,b,c), 50% CR (d,e,f) and 70% CR (g,h,i) with three steps.

The precipitates in all alloys were identified by analyzing the SADP and the chemical compositions obtained from the EDS. The analysis on the precipitates in the alloys revealed that the alloys cold-rolled by different rates and different steps mainly had two types of precipitates. Most of the precipitates, whose average size was approximately 130 nm, were identified as  $Zr(NbFe)_2$  which had a hcp crystal structure. Another type of precipitates with small size (less than 60 nm) was bcc  $\beta$ -Nb.

#### 2.4 Texture Development during Cold-rolling

From the results of Kearns factor calculation, it was found that  $f_n$  increased and  $f_r$  decreased with an increase of processing step when cold-rolling rate was kept as a constant (Fig. 3). For  $f_n$  Kearns factor, the increasing degree increased with an increase of cold-rolling rates, indicating more and more basal pole close to normal direction (ND) when the cold-rolling rate ranged from 30% to 70%.

However, it should be noted that  $f_t$  increased generally when cold-rolling rate was 30% and it decreased when cold-rolling rate was up to 70%. It meant the degree of (0002) basal pole tilting to transverse direction (TD) increased with an increase of processing steps under 30% cold-rolling, which was corresponding to the following two facts. One the one hand, the increasing degree of  $f_n$  under same cold-rolling rate was very small and nearly invariant after 2<sup>nd</sup> cold-rolling. On the other hand, the decreasing degree of  $f_r$  under 30% CR was biggest compared with those of other two rates. For 70% cold-rolling, the decrease of  $f_t$  complemented the obvious increase of  $f_n$  under the same trend of  $f_r$ . For the middle cold-rolling rate (50%),  $f_t$  was nearly invariant during different processing steps, indicating that this was a critical value for the cold-rolling rate.

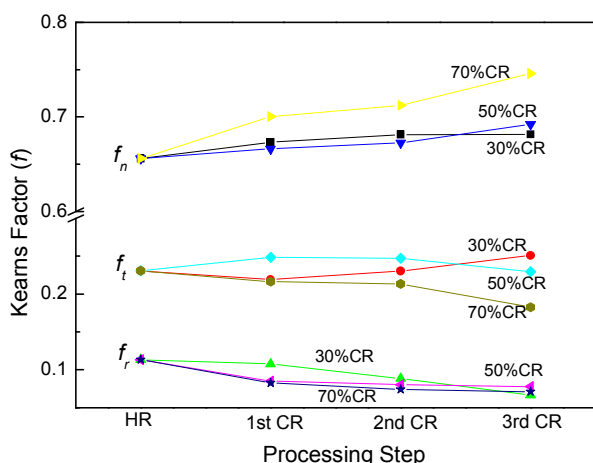


Fig. 3. The relationship between Kearns factor ( $f$ ) and rolling step of the alloy with different rolling rate.

### 3. Conclusions

In the present study, a hot-rolled Zr-1.0Nb sheet was cold-rolled and annealed by three steps and different rates. The results showed that the grain size decreased with an increase of cold-rolling rates range from 30% to 70%, and the grain size was nearly invariant under same cold-rolling rate. All samples with different treatments had two types of precipitates: hcp  $Zr(NbFe)_2$  and bcc  $\beta$ -Nb.

For higher than 50% CR, the degree of (0002) basal pole tilting to ND strengthened and TD weakened obviously with an increase of processing steps. For lower than 50% CR, the degree of (0002) basal pole tilting to ND and TD strengthened simultaneously but indistinctively with an increase of processing steps. 50% CR was a transition.

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