

# Microstructure of a Two Phase Zirconium-Niobium Alloy after Heat Treatments

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## 1. INTRODUCTION

Recrystallization in two phase alloys is something more complicate comparing with single phase alloys. In this paper, using transmission electron microscopy, we will present microstructural aspects concerning the recovery and recrystallization in a 60% cold-worked Zr-Nb alloys (Zr with 0.25wt% ; 0.5wt% ; 1.0wt% ; 1.5wt% Nb) . after isochronal heat treatments. One of the principal deal is to correlate the increasing presence of new phases in the metallic matrix (  $\alpha$  - zirconium ,  $\beta$  - zirconium and  $\beta$  - niobium ) , changing recovery and recrystallization processes and also the final microstructure ( grain size refinement , presence of fine and coarse precipitates , second phases ). The martensitic transformation from  $\beta$  phase (bcc structure) to the metastable Nb-saturated "transformed  $\alpha$ " with hcp structure is the responsible for the increasing of the mechanical properties of these alloys ( Holt 1976 ; Lee 1970 ) .

## 2. EXPERIMENTAL DETAILS

A serie of retangular sheets of 3 x 0.8 x 0.5 cm was obtained from the Zr-Nb alloy ingots. The samples were annealed at 650 °C , in an argon atmosphere , for one hour , followed by a cold water quenching and , after a chemical cleaning , the samples were 60% cold rolled (thickness) . Then , the samples were heated , also in an inert atmosphere , in the temperature range 300 - 1050 °C for one hour and cooled to room temperature . The 3 mm discs for TEM were prepared in a double jet electrolytic polishing machine using 90% methanol + 10% perchloric acid electrolyte at - 15 °C . TEM analysis of the samples were conducted at 200 kV accelerating voltage in a Jeol JEM 200C microscope (conventional imaging techniques as well as selected area diffraction techniques) . Some EDS microanalysis in TEM ( JEM 2000FXII ) were also utilised to identify the second phase .

## 3. RESULT AND DISCUSSION

The heat treatments of the cold-worked alloys promote the recovery and

recrystallization processes. The sequence of electron micrographs obtained by TEM (Figs. 1 - 3) shows this evolution. The Fig. 1a shows a microstructure of one of the as-cast Zr-Nb alloys and the Fig. 1b shows a heat-treated (650 °C for 1h) and 20% cold worked Zr-1.0wt%Nb. The electron micrograph of the Fig. 2a brings a microstructure of the recovery process on a 60% cw Zr-0.25wt%Nb after 450 °C for 1h. The microstructure of Zr-1.0wt%Nb in the Fig. 2b shows the beginning of the recrystallization process (550 °C for 1h). The electron micrograph of the figure 3a shows a microstructure of a completely recrystallized Zr-0.5wt%Nb alloy (T= 850 °C for 1h). The electron micrograph of the Figure 3b brings a duplex microstructure ( $\alpha$ -Zr +  $\beta$ -Zr with Nb dispersion) of the Zr-1.5wt%Nb heat-treated at 750 °C for 2h.

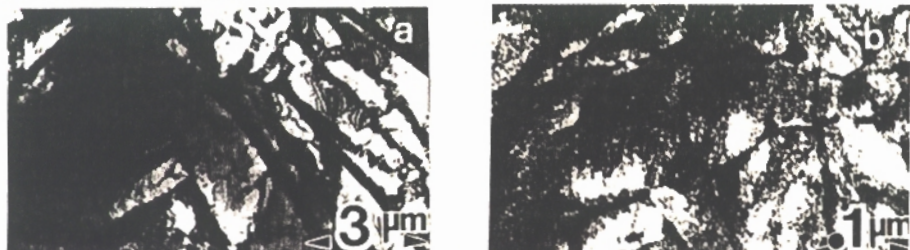


Figure 1 : a) As-cast Zr-0.5wt%Nb ;  $\alpha$ -Zr grains surrounded by  $\beta$ -Zr .  
b) Zr-1.0wt%Nb heat treated at 650 °C for 1h , 60% cold-worked .

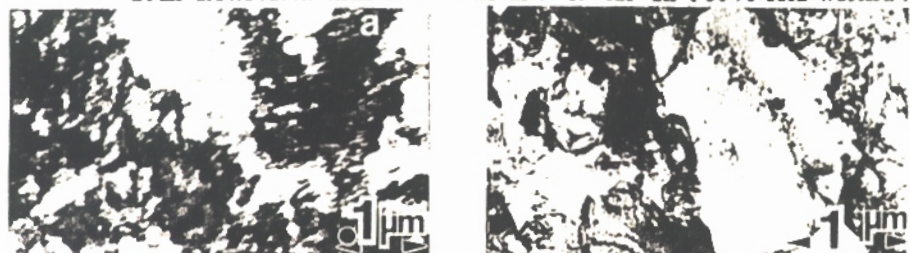


Figure 2 : a) Zr-0.25wt%Nb, 60% cw, heat treated at 450 °C for 1h; recovery process. b) Zr-1.0wt%Nb, 60% cw, heat treated at 550 °C for 1h beginning of the recrystallization process.

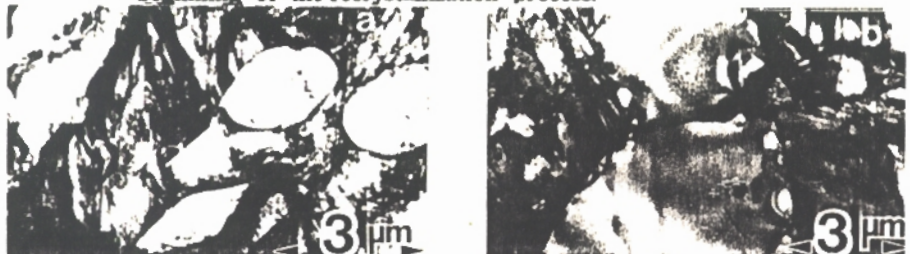


Figure 3 : a) Zr-0.5wt%Nb, 60% cw , heat treated at 850 °C for 1h ; completely recrystallized with twinned grains due to the  $\beta$ -Zr quenching ; b) Zr-1.5wt%Nb, 60% cw, heat treated at 750 °C for 2h ;  $\alpha$ -grains and " $\alpha$ -transformed " (by quenching )

Holt, R. A. , J. of Nucl. Mat. , 1976 , 59 , 234-242.

Lee, D. , J. of Nucl. Mat. , 1970 , 37 , 159-170.