

# IN-SITU CHARACTERIZATION OF LATTICE STRUCTURE EVOLUTION DURING PHASE TRANSFORMATION OF ZR-2.5NB

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## Abstract

The  $\alpha$ - $\beta$  phase transformation behavior of Zr-2.5Nb has been characterized in real time during an in-situ neutron diffraction experiment. The Zr-2.5Nb material in the current study, at room temperature, consists of  $\alpha$ -Zr phase (*hcp*) and two  $\beta$  phases (*bcc*), a Nb rich  $\beta$ -Nb phase and retained, Zr rich,  $\beta$ -Zr(Nb) phase. Other than the Burgers and Potter orientation relationships between (002) *hcp* and (110) *bcc*, this is the first time the transformation from retained Zr rich  $\beta$ -Zr(Nb) phase to  $\beta$ -Zr with increasing temperature has been reported in the literature. It is suggested that this is related to a dynamic equilibrium of the solubility of Nb atoms in the Zr *bcc* unit cells.

## Introduction

As an important nuclear reactor material, Zr-2.5Nb (wt %) has a low neutron absorption cross-section, high corrosion resistance in water and high strength and creep resistance. In thermodynamic equilibrium, it consists of  $\alpha$ -Zr phase (*hcp*) and  $\beta$ -Nb phase (*bcc*) at room temperature (Figure 1). Above the eutectoid temperature of  $T_{eu} = 610^\circ\text{C}$  the alloy consists of a mix of  $\alpha$ -Zr phase and Zr rich  $\beta$ -Zr(Nb) phase. Above the  $T_\beta = 860^\circ\text{C}$  temperature a  $\beta$  solid solution is formed. The room temperature two-phase material has been explored widely considering aspects of microstructure characterization after different heat treatments, high temperature deformation and variant selection during phase transformation. However, the underlying crystal properties, e.g. the lattice parameters and thermal expansion of the different phases during the phase transformations have not been tested in-situ and in real time. With a novel neutron diffractometer and a vacuum furnace, we recorded for the first time the

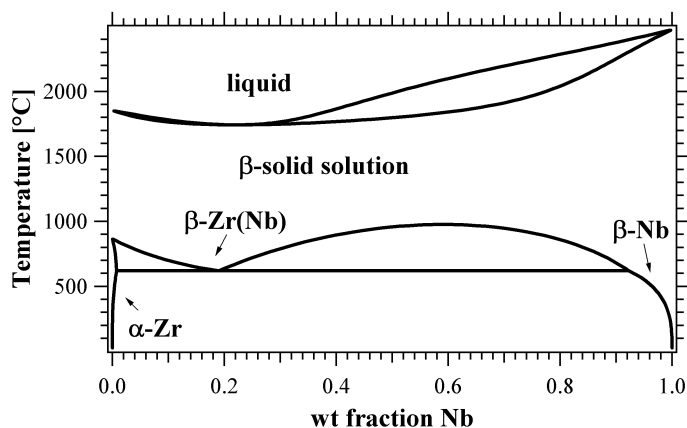


Figure 1: Phase diagram of Zr-Nb

crystalline properties of Zr-2.5Nb during the  $\alpha$ - $\beta$  phase transformation.

Table 1 Chemical compositions of the alloys (wt%)

Nb	Fe	O	Hf	Zr
2.5	0.01	0.03	0.04	Balance

## Experiment

The starting Zr-2.5Nb material was obtained from an extruded tube. The chemical analysis is given in Table 1. The material was machined to a final form consisting of cylindrical specimens of 5 mm diameter and 20 mm length.

In-situ neutron diffraction tests were conducted on the high-intensity powder diffractometer WOMBAT at ANSTO [2]. The wavelength was 1.668 Å. In this study, the specimen was heated from room temperature to 500°C at a rate of 20°C/min and from 500°C to 950°C at 10°C/min while diffracted beams were continuously recorded every 1 minute by a cylindrical area detector covering a range of 120° in-plane diffraction angle. The obtained diffraction patterns were analyzed by the Rietveld method and individual peak positions were calculated by single peak fitting.

## Results

Figure 2 shows the diffraction pattern at room temperature. In addition to the majority phase,  $\alpha$ -Zr, an amount of  $\beta$ -Nb phase and retained eutectoid composition  $\beta$ -Zr(Nb) phase with lattice parameters of  $a = 3.3016 \text{ \AA}$  and  $3.4361 \text{ \AA}$ , respectively were also detected.

Given in Figure 3 is a stacked sequence of individual

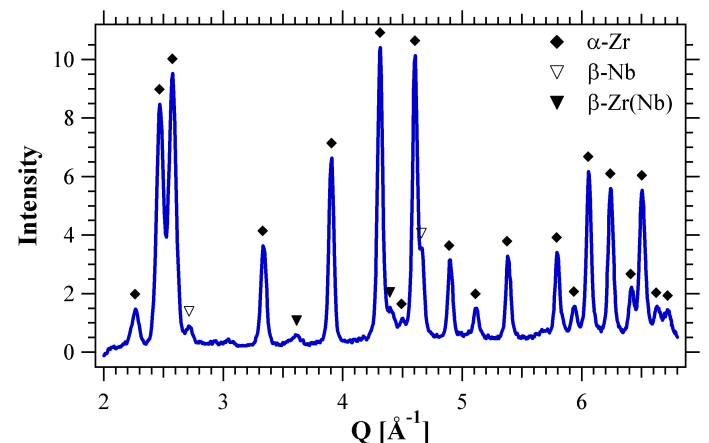
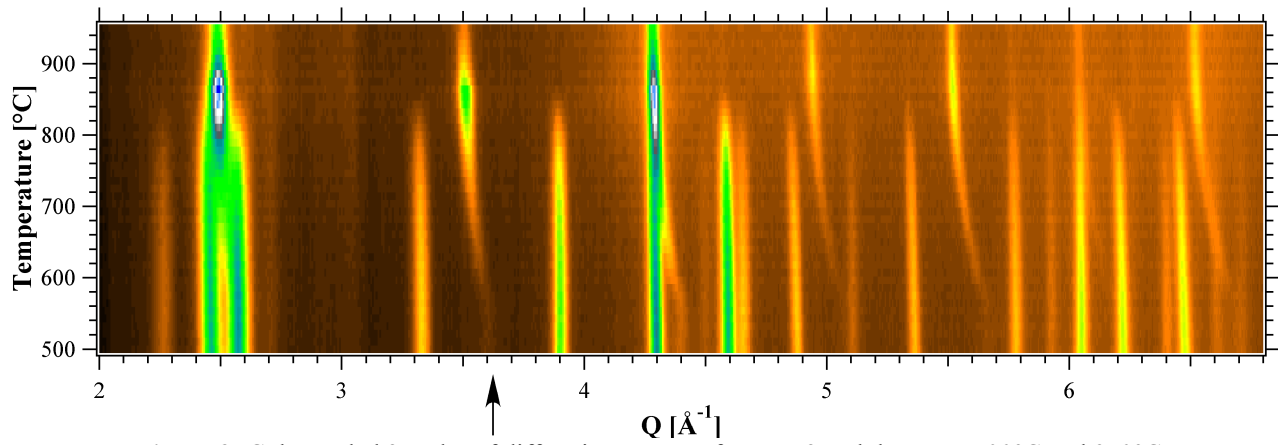


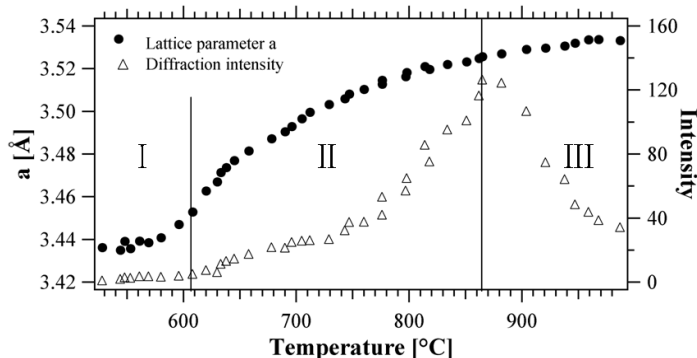
Figure 2: Diffraction pattern of Zr-2.5Nb at room temperature



**Figure 3:** Color-coded 3D plot of diffraction patterns from Zr-2.5Nb between 500°C and 950°C

diffraction patterns with the vertical axis being temperature and intensity being indicated by color. The amount of  $\beta$ -Zr(Nb) phase starts to increase above  $T_{eu} = 610^\circ\text{C}$  while  $\alpha$ -Zr vanishes totally at  $T_\beta = 860^\circ\text{C}$ ; in accordance with the phase diagram [3]. Also the Burgers / Potter orientation relation of  $(002)_{hcp} // (110)_{bcc}$ , which has been shown to occur in the allotropic transformation of  $hcp$ - $bcc$ - $hcp$ , is observed in this 3D diffraction plot of Zr-2.5Nb. Additionally, the effect of the alloy element Nb on this  $hcp$ - $bcc$  transformation process can be investigated. The arrow in Figure 3 locates the 200 peak of retained  $\beta$ -Zr(Nb). During the phase transformation the  $\beta$ -Zr(Nb) peak shifts to the position of the expected  $\beta$  solid solution peak.

The evaluated lattice parameter together with the peak intensities are plotted in Figure 4. The  $\beta$ -phase lattice parameter appears to experience three distinct lattice expansion stages, marked as I, II and III. From 500°C to  $T_{eu}$ , it increases from 3.4361 Å to 3.4529 Å, while from  $T_{eu}$  to  $T_\beta$ , it increases significantly from 3.4529 Å to 3.5257 Å. Above  $T_\beta$  it gradually reaches its asymptotic value of 3.5331 Å at 950°C.



**Figure 4.** Lattice parameter and diffraction intensity of  $\beta_{200}$  during phase transformation

The strong changes in lattice parameter testify to variations in the Nb concentration of the  $\beta$ -Zr(Nb) phase during the  $\alpha$ - $\beta$  transformation. Such large lattice changes cannot be attributed to interface strain, intergranular strains or thermal strains alone due to their magnitude.

Below the eutectoid temperature, the alloy consists of  $\alpha$ -Zr,  $\beta$ -Nb and some retained  $\beta$ -Zr(Nb). When the temperature exceeds the eutectoid point the concentration of Nb in  $\beta$ -Zr(Nb) decreases. At the same time, the transformation  $\alpha$ -Zr  $\rightarrow$   $\beta$ -Zr(Nb) commences as seen by the increase of intensity in Figure 4. The dynamic balance between the phases is driven by the change of temperature until the final single  $\beta$  solid solution of composition Zr-2.5Nb is achieved. This process can also be followed in the other  $\beta$ -Zr(Nb) diffraction peaks. The occurrence of metastable omega phase with a near- $hcp$  structure below 500°C is also observed, providing a supplementary role in the lattice changes associated with the  $\beta$ -Zr(Nb) phase.

## Conclusions

The current work provides a method of analyzing phase transformation processes in-situ and in real time. Evolutions of lattice parameters elucidate the changes of atomic concentrations during the phase transformation; which are dramatic during the eutectoid reaction in the Zr-2.5Nb system. Quantitative analysis of the concentrations and other yet unpublished details can be extracted from the data, such as the nucleation of  $\omega$  phase below 500°C as well as the intensity decrease above  $T_\beta$ , due to grain recovery and growth. This will be reported elsewhere.

## References

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