

# The Influence of Different Contents of Bi Addition on the Corrosion Behavior of Various Zirconium-Based Alloys

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## Abstract

Zr-4(Zr-1.5Sn-0.2Fe-0.1Cr, wt%), S5(Zr-0.8Sn-0.34Nb-0.39Fe-0.1Cr), T5(Zr-0.7Sn-1.07Nb-0.32Fe-0.08Cr) and Zr-1Nb were adopted to prepare Bi-containing zirconium alloys for systematically investigating the effect of Bi addition on the corrosion resistance of zirconium alloys. The specimens were corroded in superheated steam at 400°C/10.3 MPa, and in lithiated water with 0.01 M LiOH or in deionized water at 360°C/18.6 MPa by autoclave testing. Results show that the corrosion resistance increases with the increasing of Bi content dissolved in  $\alpha$ -Zr. But the presence of Bi-containing second phase particles (SPPs) is unfavorable for the enhancement of corrosion resistance. This indicates that the Bi dissolved in  $\alpha$ -Zr matrix plays an important role in improving the corrosion resistance, while the precipitation of the Bi-containing SPPs does harm to the corrosion resistance.

## Keywords

Bi, Zirconium Alloy, Corrosion Resistance, SPPs

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## 1. Introduction

Zirconium-based alloys have been used as fuel cladding materials in pressurized water reactors due to their low cross-section for thermal neutron absorption, good corrosion resistance and adequate mechanical properties. However, the waterside corrosion resistance of the fuel cladding affects the service life of high burn-up fuel as-

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semblies. The higher demand for burn-up and refueling period in reactor requires new zirconium alloys with advanced performances [1]-[4].

There are three series (Zr-Sn, Zr-Sn-Nb and Zr-Nb) of promising zirconium alloys, such as Zr-4 (Zr-1.5Sn-0.2Fe-0.1Cr, wt%) (Zr-Sn serie), S5 (Zr-0.8Sn-0.34Nb-0.39Fe-0.1Cr) (Zr-Sn-Nb serie), T5 (Zr-0.7Sn-1.07Nb-0.32Fe-0.08Cr) (Zr-Sn-Nb serie) and Zr-1Nb (Zr-Nb serie). Optimizing composition of zirconium alloys is a common method to develop new alloys with excellent corrosion resistance [5] [6]. Many researches showed that the influence of alloying elements on the corrosion resistance depended on the concentration of alloying elements in  $\alpha$ -Zr matrix: the corrosion resistance of zirconium alloys was improved with the increase of the content of alloying elements (Cu, Fe, Cr) dissolved in  $\alpha$ -Zr [7]-[12]. It indicates that alloying elements with relative high concentration in  $\alpha$ -Zr are possible alloying elements of zirconium alloys from consideration of their solid solute content dissolved in  $\alpha$ -Zr. The solubility of many alloying elements are very low (the maximum solubility of 120  $\mu$ g/g at 700°C for Fe and the maximum solubility of 0.1 wt% at 580°C for Cu), while the solubility of Bi in  $\alpha$ -Zr is 6 wt% at 580°C according to Zr-Bi phase graph [13]. Bi is also a suitable alloying element from consideration of capture cross-section for thermal neutrons. And it was reported that adding 0.1 wt% to 0.5 wt% Bi can improve the corrosion resistance of Zr-1Nb alloy [14]. So far, there is no systematic research on Bi. From the consideration of the solubility and cross-section for thermal neutron absorption, our group studied the effect of Bi on the microstructures and corrosion resistance of various zirconium alloys. Zr-4, S5, T5 and Zr-1Nb were adopted as matrix alloys to prepare the zirconium alloys containing Bi of 0.05 wt% - 0.5 wt%. The microstructure of these zirconium alloys and part of corrosion results have been reported in [15]-[19]. The corrosion resistance of these Bi-containing zirconium alloys was systematically reported to comprehensively understand the influence law of the different contents of Bi addition on the corrosion behavior of various zirconium-based alloys in this study.

## 2. Materials and Experiments

Four series of Bi-containing alloys were prepared by adding 0.05 wt% - 0.5 wt% Bi into Zr-4, S5, T5 and Zr-1Nb matrix alloys, denoted as Zr-4 + xBi ( $x = 0.1, 0.3, 0.5$ , wt%), S5 + xBi ( $x = 0.1, 0.3, 0.5$ ), T5 + xBi ( $x = 0.1, 0.3, 0.5$ ) and Zr-1Nb + xBi ( $x = 0.05, 0.1, 0.2, 0.3$ ), respectively. The composition and preparation procedures of experimental alloys have been reported in [19] in details. As an aid to the readers, the preparation procedures of experimental alloys are shown in Figure 1. Alloy ingots of about 65 g were smelted by vacuum non-consumable arc smelting. In order to homogenize the composition of alloys, each ingot was turned over and smelted for 6 times. The ingots were made into plates with about 0.7 mm in thickness after they underwent hot-pressing at 700°C, homogenizing annealing at 1030°C for 40 min, hot-rolling at 700°C,  $\beta$ -quenching at 1030°C for 40 min, cold-rolling and final annealing at 580°C for 5 - 50 h. Last two steps were different for the four series of alloys. Procedure A is for T5 + xBi, S5 + xBi and Zr-4 + xBi alloys, and procedure B is for Zr-1Nb + xBi alloys.

The specimens were chemically polished using a mixed acid solution (30 vol% H<sub>2</sub>O + 30 vol% HNO<sub>3</sub> + 30 vol% H<sub>2</sub>SO<sub>4</sub> + 10 vol% HF) before every annealing. The corrosion tests were conducted in superheated steam at

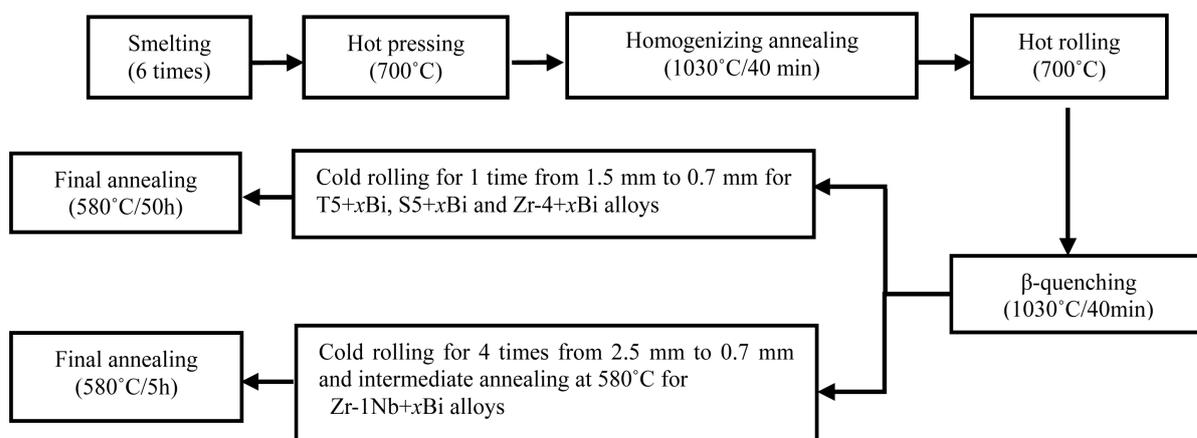


Figure 1. Preparation procedures of experimental alloys.

400°C/10.3 MPa, and in lithiated water with 0.01 M LiOH or in deionized water at 360°C/18.6 MPa by static autoclave testing, according to ASTM G 2/G 2M-06. The corrosion resistance was evaluated by measuring the weight gain per unit surface area in relation to the exposure time. The reported weight gain was a mean value obtained from 3 - 5 specimens. Before the corrosion test, the specimens were cleaned and pickled in a mixed acid which has been mentioned above, sequentially rinsed in cold tap water, boiling deionized water and then blow-dried with warm air.

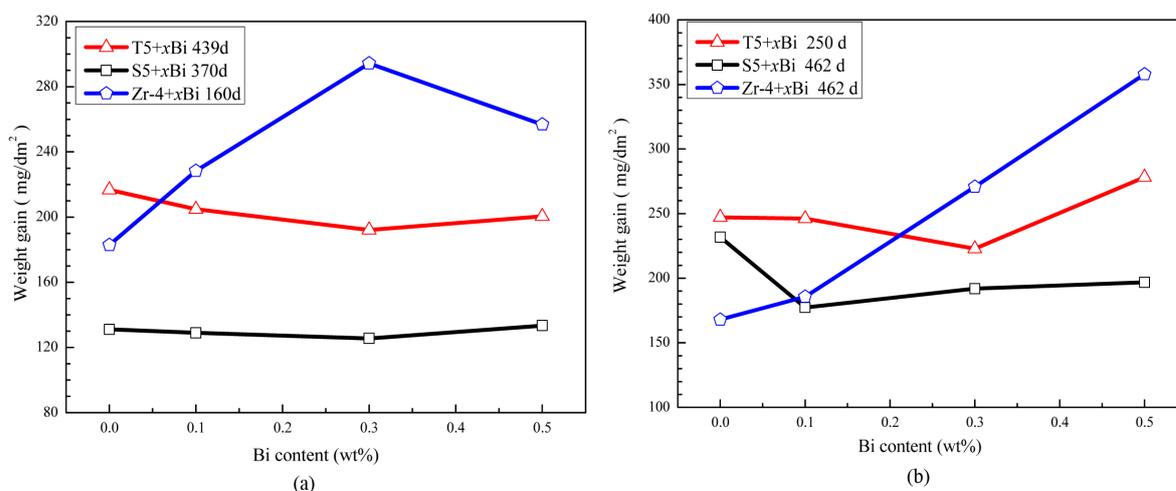
### 3. Results and Discussion

#### 3.1. Results

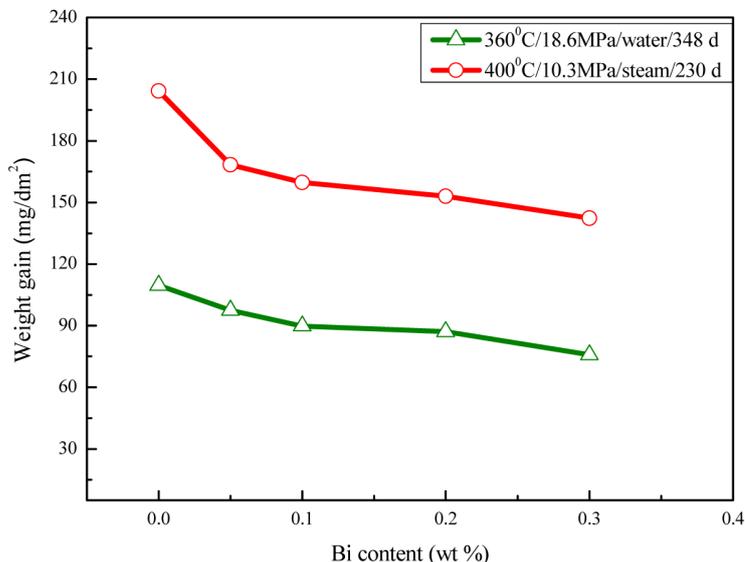
The final corrosion weight gains of Zr-4 + xBi, S5 + xBi and T5 + xBi alloys vs. Bi content in lithiated water with 0.01 M LiOH at 360°C/18.6 MPa and in superheated steam at 400°C/10.3 MPa are shown in **Figure 2**. It can be seen that the addition of 0.1% - 0.5% Bi to Zr-4 is harmful to the corrosion resistance and the variation of Bi contents has a remarkable effect on the corrosion resistance in both corrosion conditions; the corrosion resistance of the Zr-4 + xBi alloy markedly decreased with the increase of Bi content, especially in superheated steam. The addition of 0.1% - 0.5% Bi to S5 has a little beneficial effect on the corrosion resistance in lithiated water, and it can also improve the corrosion resistance in superheated steam, but the improvement effect decreases with the increasing Bi content. Adding 0.1% - 0.5% Bi to T5 can also improve the corrosion resistance in lithiated water, while in superheated steam, the addition of not more than 0.3% Bi can enhance the corrosion resistance, but adding 0.5% Bi is deleterious to the corrosion resistance. Other than the above alloys, 0.05% - 0.3% Bi addition exhibits a beneficial effect on the corrosion resistance of Zr-1Nb alloy and the corrosion resistance is markedly improved with the increase of Bi content whether in deionized water at 360°C/18.6 MPa [15] or in superheated steam at 400°C/10.3MPa (**Figure 3**) [20]. This illustrates that the effect of Bi addition on the corrosion behavior of various zirconium-based alloys depends on the original compositions of alloys and corrosion circumstances.

#### 3.2. Discussion

Our group has reported the microstructure of the above Bi-containing zirconium alloys in detail in [15]-[19]. The results are described in brief as follows: the maximum Bi content dissolved in  $\alpha$ -Zr matrix (denoted as  $C_{Bi,max}$ ) was different for various zirconium-based alloys according to appearing Bi-containing SPPs: less than 0.1% for the Zr-4 + xBi alloys, 0.1% to 0.3% for the S5 + xBi alloys, 0.3% to 0.5% for the T5 + xBi alloys, and more than 0.3% for the Zr-1Nb + xBi alloys (The data in **Table 1** are taken from our previous study [19]). According to the Zr-Bi binary phase diagram, the Bi solubility in  $\alpha$ -Zr is about 6 wt% at 580°C [13]. Therefore, there should not



**Figure 2.** Final corrosion weight gains of Zr-4 + xBi, S5 + xBi and T5 + xBi alloys vs. Bi content in lithiated water at 360°C/18.6 MPa (a) and in superheated steam at 400°C/10.3 MPa (b).



**Figure 3.** Final corrosion weight gains of Zr-1Nb + xBi alloys vs. Bi content in deionized water at 360°C/18.6 MPa and in superheated steam at 400°C/10.3 MPa [20].

**Table 1.** The information of SPPs in the alloys [19].

Alloy	Information of SPPs			
	Remelted	0.1 Bi	0.3 Bi	0.5 Bi
Zr-4 + xBi (Sn: 1.5%)	h-Zr(Fe,Cr) <sub>2</sub>	h-Zr(Fe,Cr) <sub>2</sub>	h-Zr(Fe,Cr) <sub>2</sub>	Zr-Fe-Sn-Bi
		Zr-Fe-Sn-Bi	Zr-Fe-Cr-Sn-Bi	Zr-Fe-Cr-Sn-Bi Zr-Fe-Cr-Bi
S5 + xBi (Sn: 0.8%)	Zr(Fe,Cr,Nb) <sub>2</sub>	h-Zr(Fe,Cr,Nb) <sub>2</sub>	h-Zr(Fe,Cr,Nb) <sub>2</sub>	h-Zr(Fe,Cr,Nb) <sub>2</sub>
			o-Zr(Bi,Sn,Fe) <sub>2</sub>	o-Zr(Bi,Sn,Fe) <sub>2</sub> Zr-Fe-Cr-Nb-Sn-Bi
T5 + xBi (Sn: 0.7%)	h-Zr(Fe,Cr,Nb) <sub>2</sub>	h-Zr(Fe,Cr,Nb) <sub>2</sub>	h-Zr(Fe,Cr,Nb) <sub>2</sub>	h-Zr(Fe,Cr,Nb) <sub>2</sub>
				o-Zr(Fe,Sn,Bi) <sub>2</sub> Zr-Fe-Cr-Nb-Sn-Bi
Zr-1Nb + xBi (Sn: 0%)	$x \leq 0.3\%$ , none of Bi-containing SPPs was detected, and there were only $\beta$ -Nb and h-Zr(Nb,Fe,Cr) <sub>2</sub>			

be Bi-containing SPPs in the alloys with Bi content not more than 1%. However, the Bi-containing SPPs were really detected in the experimental alloys in the Bi-containing zirconium alloys in this study. Moreover, the addition of Bi promotes the precipitation of Sn as SPPs. The content of Bi dissolved in  $\alpha$ -Zr matrix increases with the decrease of Sn content in the alloys. The reason has been discussed in [16] [19] in detail.

The effect of Bi addition on the corrosion behavior of various zirconium-based alloys is related to the original compositions of alloys and corrosion conditions. The optimizing content of Bi corresponding to the corrosion resistance is less than 0.1% for Zr-4, 0.1% to 0.3% for S5, 0.3% for T5 and 0.3% for Zr-1Nb alloy based on this study, and the content of Bi is 0.1% for Zr-4, 0.3% for S5 and 0.5% for T5 when the Bi-containing SPPs are

precipitated. It indicates that the Bi dissolved in  $\alpha$ -Zr matrix is beneficial for improving the corrosion resistance of zirconium alloys, and the precipitation of the Bi-containing SPPs is harmful to the corrosion resistance.

When zirconium alloys are oxidized, the SPPs and the alloying elements dissolved in  $\alpha$ -Zr will also be oxidized, which affects the characteristics of oxide film, and ultimately influences the corrosion resistance of alloys. The oxidation of alloying elements dissolved in  $\alpha$ -Zr on the corrosion resistance of alloys has dispersive effect than that of SPPs; Moreover, Bi dissolved in  $\alpha$ -Zr can be more likely to dissolve in  $ZrO_2$  than the Bi in SPPs, which can reduce the decrease of surface free energy due to the infiltration of oxygen ions and hydroxide ions. Therefore, the vacancy in the oxide film cannot easily diffuse and condense to form the pore-clusters and cracks, and the corrosion resistance of zirconium alloys can be improved [21].

The addition of Bi lets Sn precipitate from  $\alpha$ -Zr matrix as SPPs. As the Pilling-Bedworth of Zr, Bi and Sn are 1.56, 2.27 and 1.32 [22], respectively, there will be major additional stresses when the SPPs are oxidized, which can promote the formation of pores and micro-cracks and provide more diffusion paths for oxygen ions and hydroxide ions, and eventually accelerate the corrosion of alloys. At the same time, the precipitation of SPPs containing Bi and Sn can counteract the improving effect of Bi dissolved in  $\alpha$ -Zr on the corrosion resistance of zirconium alloys. In addition, the precipitation of SPPs containing Bi and Sn also decreases the content of Sn dissolved in  $\alpha$ -Zr. It has been reported that Sn can offset the harmful effect of nitrogen in zirconium alloys, and too high or too low Sn content would produce disadvantageous effect on the corrosion resistance of zirconium alloys [22]. This may be the reason that the precipitation of SPPs containing Bi and Sn is harmful to improve the corrosion resistance of zirconium alloys.

From another angle, the corrosion of zirconium alloys in deionized water or in superheated steam with high temperature and pressure is also an electrochemical process. The SPPs at the Zr/ $ZrO_2$  interface and  $\alpha$ -Zr matrix constitute many micro batteries during oxidation, and  $\alpha$ -Zr matrix is preferentially oxidized as anode [23]. On one hand, the Bi dissolved in  $\alpha$ -Zr matrix may change the potential of matrix, and reduce the potential difference between the matrix and SPPs, which will slow down the process of electrochemical corrosion and improve the corrosion resistance of alloys; On the other hand, the addition of Bi change the composition and type of SPPs, and the potentials of various SPPs exist diffidence. Therefore, the electrochemical behavior of the system will be changed and the corrosion resistance of zirconium alloys will be affected.

Many researches thought that the concentrations of alloying elements dissolved in  $\alpha$ -Zr matrix were the main factor to affect the corrosion resistance of zirconium alloys [9]-[12] [24] [25]. Other researches considered the SPPs to be the main factor [4] [26] [27]. In this study, it is obvious that the Bi dissolved in  $\alpha$ -Zr matrix plays a beneficial role in the corrosion resistance, while the Bi-containing SPPs precipitated from the matrix plays a harmful role in the corrosion resistance. This further illustrates that a suitable increase in concentration of alloying elements in  $\alpha$ -Zr matrix is of benefit to improving the corrosion resistance of zirconium alloys [9]-[12] [24].

## 4. Conclusion

The effect of Bi addition on the corrosion behavior of various zirconium-based alloys depends on the compositions of mother alloys and corrosion conditions. The solid solution of Bi in  $\alpha$ -Zr matrix plays a beneficial role in the corrosion resistance of zirconium alloys, while the precipitation of the Bi-containing SPPs plays a harmful role in the corrosion resistance of zirconium alloys.

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