



# Effect of yttrium on nucleation and growth of zirconium hydrides



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## ABSTRACT

Addition of yttrium in zirconium causes precipitates of yttrium, which form two types of particles and are oxidized upon heat treatment. One type of particles with sub-micrometer scale sizes has a low population, whereas the other with nano scale sizes has a high population and cluster distribution. Owing to strong affinity of yttrium to hydrogen, the nanoparticles, mostly within the grains of the Zr–Y alloy, attract nucleation of hydrides at the clusters of the nanoparticles and cause preferential distribution of intragranular hydrides. In comparison with that of Zr, additional nanoparticles in the Zr–Y alloy impede further growth of hydride precipitates during hydriding. It is deduced that the impediment of growing hydride precipitates by the nanoparticles is developed during an auto-catalytic nucleation process, which leads to formation of thin and intragranular hydrides, favorable to mitigation of hydride embrittlement.

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## 1. Introduction

Zr based alloys have been extensively used as structural components in nuclear reactors, such as clad tubes, due to their superior mechanical properties, corrosion resistance and neutron transparency [1]. The pickup of excess hydrogen into Zr based alloy claddings in service could lead to precipitation of zirconium hydrides [2,3]. These hydride precipitates are brittle in nature, and are likely to cause stress-concentration at the edges of the platelets [4,5]. The loss of both ductility and fracture toughness due to a high population of hydrides, i.e., hydride embrittlement (HE) [6,7] as well as delayed hydride cracking (DHC) [8], are two principal hydride associated problems, which degrade the mechanical properties of the Zr based alloy claddings [9].

Zirconium hydrides, as cracking initiators in Zr based alloys, have attracted significant attention in the last few decades [10–24]. It has been identified that the degree of HE depends on the tested temperature and the microstructure of the material [18,25], in particular, the quantity [11], orientation [17,26] and morphology [27] of the hydrides in the material. Therefore, it is reasonable to modify one or more of these parameters to mitigate HE. Alloying, such as adding elements of niobium [28] and yttrium [29,30] into the alloys, is a rational approach to modify hydride morphology and eliminate the susceptibility of the claddings to HE. In some recent studies, Batra et al. [29,30] have shown that adding a small amount of yttrium into zirconium alloys and implementing certain heat treatments (mainly quenching) could provide

denser sites for nucleation of hydrides and reduce hydride precipitates to smaller size, hence mitigate HE to some extent. However, the microstructural modification of the alloys by heat treatments and its effects on nucleation and growth of zirconium hydrides have not been adequately investigated in the studies. Therefore, we have carried out some further studies on the hydrides of a prior recrystallized Zr–Y alloy based on the approach, and reported the observation and relevant analysis in this paper. The objective of the paper is to provide some more detailed information and mechanism on the influence of alloying yttrium on structural modification and nucleation and growth of the hydrides in Zr–Y alloys.

## 2. Experimental procedures

Zr and Zr–Y (Zr–0.8 wt.%Y) ingots were prepared by arc melting nuclear grade sponge zirconium (99.9%) and high-purity yttrium pieces (99.9%) under argon atmosphere. The ingots were remelted several times to ensure chemical homogeneity. The ingots were then sealed separately in quartz tubes and heated to selected temperatures for 30 min and water quenched by breaking the hot quartz tubes in water. The heating temperature of 1000 °C and 1160 °C were selected for Zr and Zr–Y ingots to ensure formation of  $\beta$ -Zr phase and yttrium solution, respectively. The ingots were then hot-rolled to 4 mm thick after a preheating at 580 °C for 20 min and subsequently cold-rolled two times to a final thickness of 0.8 mm. The sheets were intermediate-annealed at 580 °C for 4 h between the above rolling processes. The final cold-rolled sheet samples of Zr metal and Zr–Y alloy were heated up to 580 °C for 6 h to reach fully recrystallized form of  $\alpha$ -Zr metal and alloy. The

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**Table 1**  
Chemical compositions of Zr metal and Zr–Y alloy samples.

Materials	Chemical compositions		
	Y (wt.%)	O (wppm)	Zr
Zr	–	400	Bal.
Zr–Y	0.76	395	Bal.

chemical compositions of the recrystallized samples were determined by inert gas melting method and listed in Table 1.

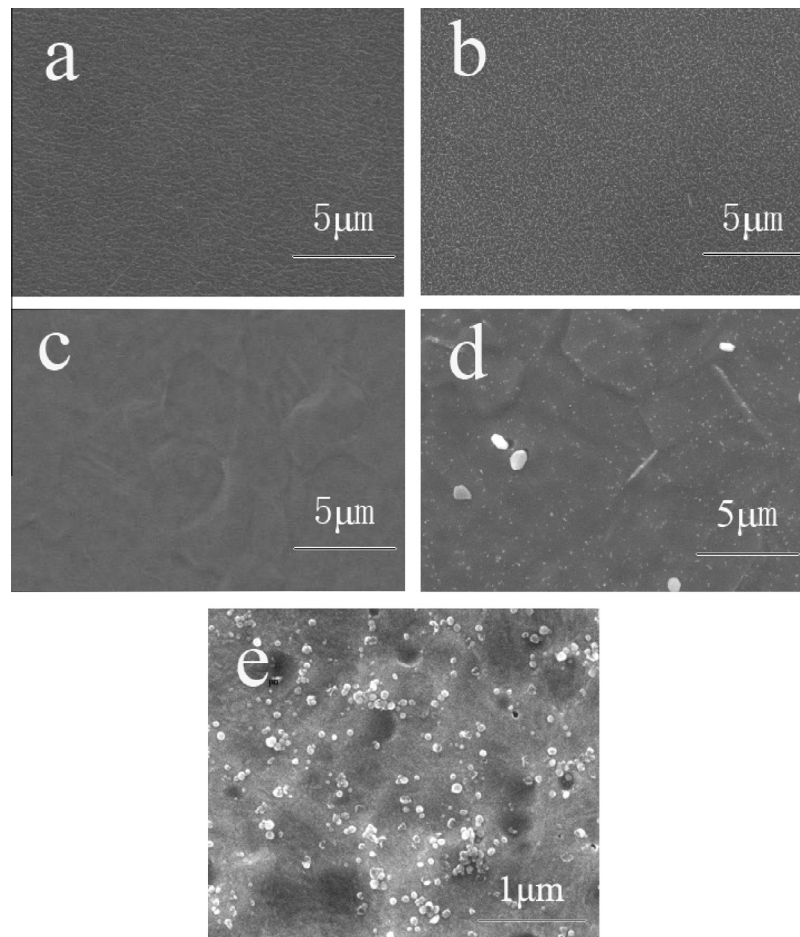
The hydriding treatment was performed in a self-designed gaseous hydrogen-charging device. Before hydriding, the recrystallized samples were mechanically polished to remove thin oxide films on the surfaces. During hydriding, the samples were heated to a selected hydrogen pickup temperature of 350 °C, and cooled to room temperature in furnace. The hydrogen concentrations of about 300 wppm in the samples were controlled by the hydrogen pressure in the sample chamber (the accurate hydrogen concentrations of the Zr and Zr–Y samples were confirmed as 306 wppm and 294 wppm, respectively). The hydrided samples were treated by acid etching in a solution of 45% HNO<sub>3</sub>, 45% H<sub>2</sub>O and 10% HF and subsequently washed in water and ethanol before examinations of Optical Microscopy (OM, LEICA MEF4A), Scanning Electron Microscopy (SEM, FEI Inspect F50) and attached Energy Disperse Spectroscopy (EDS). The recrystallized samples without hydriding were also treated by the same procedures for SEM and EDS examinations. The phase composition of the recrystallized Zr–Y alloy

was characterized by X-ray diffraction (XRD, RIGAKU D/max-2500PC) with Cu K $\alpha$  radiation.

### 3. Results

Fig. 1 shows SEM micrographs of the water-quenched and the recrystallized Zr and Zr–Y alloy samples. There is no obvious difference between the morphologies of the water-quenched Zr and Zr–Y alloy samples (see Fig. 1a and b). The micrographs of both water-quenched Zr and Zr–Y samples show typical single phase morphology, confirming the formation of yttrium solution Zr–Y alloy after quenching treatment. However, in comparison with the recrystallized Zr sample (see Fig. 1c), the recrystallized Zr–Y alloy sample presents a multiphase morphology, where two types of precipitated particles different in their sizes and compositions can be identified on the matrix of the alloy (see Fig. 1d). The large bright precipitated particles with a spherical shape and a sub-micrometer scale size of around 500 nm have a low population and do not show obvious cluster distribution. On the other hand, there are a large number of dark sphere particles, which have much smaller size of nanometer-scale, and are distributed in clusters (see Fig. 1d and e). As shown in Fig. 1d and e, most of these nanoparticles are within the grains of the Zr–Y alloy and no obvious preferential distribution of the nanoparticles on the grain boundaries is observed.

The precipitated particles observed in the recrystallized Zr–Y alloy sample are further characterized by relevant EDS and XRD



**Fig. 1.** SEM micrographs of water-quenched (a) Zr and (b) Zr–Y alloy; and recrystallized (c) Zr and (d) Zr–Y alloy; and (e) clusters of nanometer scale particles in recrystallized Zr–Y alloy.

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