ESRF	**Experiment title:** In-situ hydrogen charging of a Zirconium powder to study iso-thermal precipitation of hydrides	**Experiment number**: MA-1371
Beamline: ID15-B	**Date of experiment**: from: 2011-11-23 to: 2011-11-26	**Date of report**: 2012-02-21
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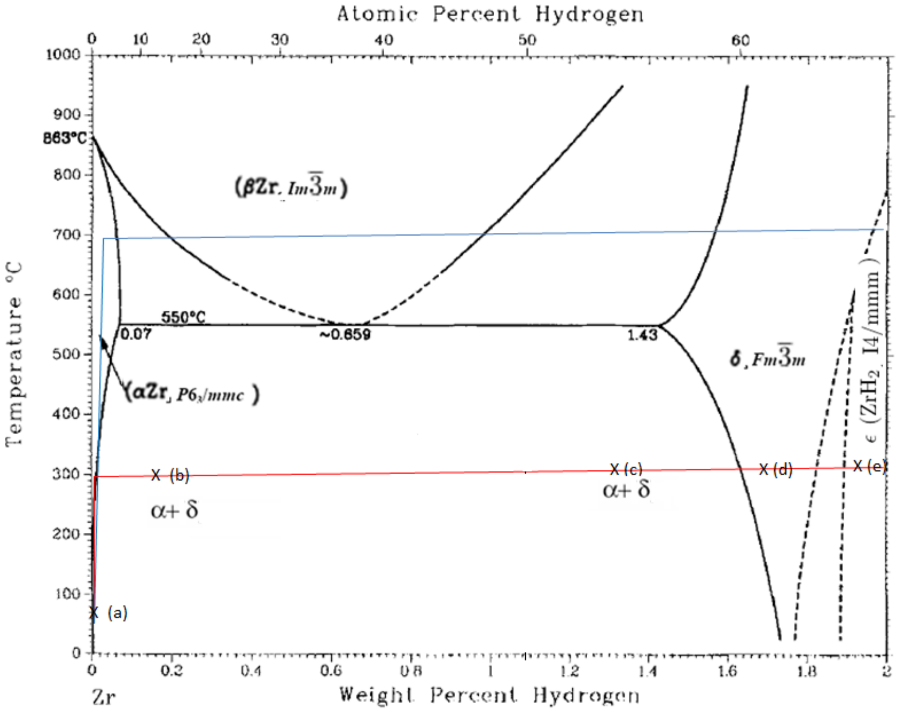
Report: Zirconium and its alloys have good mechanical property with low hydrogen absorption cross section. So they wiedly used as construction materials at atomic reactors. However, Zr have a strong affinity for hydrogen which leads to hydrogen pick-up during a corrosion reaction when exposed to water. The hydrogen is readily in solution at higher temperature but precipitates as Zirconium hydrides at ambient temperatures. At least three phases are known to exist at ambient temperature depending on hydrogen concentration and quenching rate. Even Zr-hydrides have been known and studied for several decades still some mechanisms are largely misunderstood, owing to the particular nature of hydrogen and its high diffusivity even at low temperature.

During the experiment carried out at ESRF at beam line ID15-B we have attempted to:

1. Collect a high resolution data sets for structural analysis and modelling work.
2. Proceed a in-stiu hydrogen charging to study the kinetiks of precipitation and the transition between the epsilon and delta hydride phases.
3. The effect of the alloying elements to the transformation and stability.

To achieve these goals, pure Zr powder brought to the ESRF and placed in quartz capillaries in an glove box in orther to avoid any oxygen contamination. To desolve any hydride which formed earlier or during preparation, all sealed cells which contains Zr powder were baked in vacuum at 1000 ^{o}C about 7-8 hours. After the heat treatment we obtain pure Zr-phase (Fig.2-a). We conducted a number of measurments on both powder and polycrystals. The two of our typical experimental setup schematically shown on phase diagram [1] with blue and red lines (Fig.1). On all experiment, the measurment were started from room temperature to then heated to 300 or 700 ^{o}C. When final temperature was reached we kept it fixed for 30 minutes, then let it back to starting temperature. The heating and cooling rate were both kept at 10ºC/min. The mass spectrometer installed on the set up was used to detect any dehydration during the post-charging heat treatments. Since the capillary inner diameter was ~2 mm, we selected 0.3x0.3 mm as beam size to ensure good diffraction signal from the specimens. Initially, we planned to carry out the charging around 100 bars but found that the reactions were too fast. So we limit the partial H_2-pressure to 0.5-10 bars. The SXRD (Synchrotron X-ray diffraction) pattern during hydrogen charging and heat treatment of the samples recorded as a function of time. Based on pre analysis of collected data, we had used fast and slow data acquisitions.



In Fig.1 the x on red line represents five different positions which belong to different phase regions, and Fig.2 shows the respective diffraction patterens for these positions. Because of limited space, the example positions and fitted diffraction pattaren for blue curve is omitted. In Fig.2 the blue line is based on real data and red is the initial Rietveld fit. From Fig.2 it can be seen that we not only able to collect continuous data for every single phases, including mixed regions during heating and cooling, but we were also able to produce accurate Rietveld fit to extract the cell parameters. In this sense we reached our first two aims. However, because of very limited time and some technical difficulity we couldn't able to reach the third aim.

To sum up, the experiment was very promising and the results will verify the discripency about the transition and stability of Zr-hydride phases for the first time using modern, high-resolution synchrotron powder diffraction techniques with in situ hydrogen loading system.

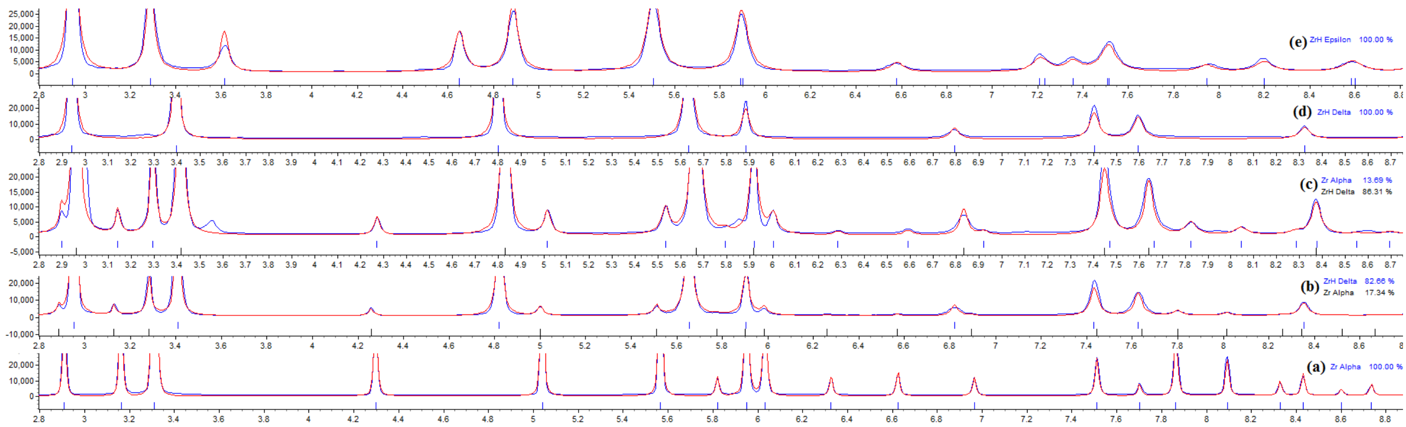


Fig.2 The selected diffraction patterns with Rietvel fit. The blue is original data from experiment and red is the Rietveld fit. The measured sites of a-b are shown in Fig.1.

Reference

[1] Zuzek E, Abriata JP, San-Martin A, Manchester FD. H-Zr (Hydrogen-Zirconium). Phase Diagrams of Binary Hydrogen Alloys. ASM International, 2000.