| ESRF | **Experiment title:** Mechanism of the alpha-omega transition in Zr studied with time-resolved X-ray absorption spectroscopy | **Experiment number**: HC-1607 |
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| **Beamline**: ID24 | **Date of experiment**: from: 6/05/2015 to: 12/05/2015 | **Date of report**: 15/02/2016 |
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## Report:

The aim of this experiment was to characterize with a time-resolved technique (ms scale) the pressure-induced alpha-omega phase transition in zirconium: kinetics, mechanism with possible intermediate phases. The kinetics of this transition has been reported to be slow, with transformation times which can reach several minutes for the direct alpha-omega transition [1], and no reverse transition under ambient temperature.

The high pressure device used in this project is a dynamic diamond anvil cell, which allows the pressurizing of a sample with pressure ramps of up to 50 GPa/ms. The principle of this device is to fill the diamond anvil cell membrane dynamically with a high velocity electric valve. This valve is synchronized with x-ray absorption spectroscopy (XAS) data acquisition, as well as time-resolved ruby luminescence spectrocopy to measure the pressure. This yields: Pressure on the sample vs $(t-t_0)$ and XAS spectra vs $(t-t_0)$, starting form the opening of the electric valve at $t_0$, with a typical time resolution of 0.2 to 1 ms.

Prior to the dynamic experiment, the transformation pressure at ambient temperature has been determined under quasi-hydrostatic loading (neon pressure medium) with the same samples (Zr foil 99.9+% purity, cutted with a fs laser), under static compression: the transformation occurs at 11.5 GPa.

We have therefore targeted this pressure range for the dynamic experiments. Ramps with 1 to 5 GPa/s have been applied to samples prepared from the same foil with the same technique. They were quasi-hydrostatically compressed in neon pressure medium. The sample was characterized with XAS before and after the pressure ramping, and during the ramp. We used nano-polycrystalline diamond anvils, which do not create any XAS parasitic signal due to Bragg diffraction [2].

The beam time has been used as followed: wavelength selection (Zr k-edge, 18.0 keV), focusing of X-rays, mounting of the time-resolved pressure measurement setup and alignment on the X-ray beam in the first two days; data acquisition (loading of one fresh Zr sample in neon pressure medium, alignment on X-ray beam, alignment of pressure measurement line, tests, one shot) in the next four days. We have been able to characterize the alpha-omega transition 8 times on 8 different samples, which all yielded similar results.

Examples of XAS spectra and pressure ramp are presented in **Figure 1.** Alpha and omega phases can be easily distinghised by their XAS spectra around 50 eV above the absorption jump. XAS spectra show that the transformation takes place at 12 GPa, a pressure which is very close to the static transformation one. The transformation is complete in less than 2 ms, which is much shorter than reported in Ref. [1]. This can be due to the fact that the loading in non-hydrostatic in Ref. [1] and quasi-hydrostatic in our experiments. In addition, the alpha-omega transition is sensitive to the impurities in the sample, which were different in the two studies. The measured tim of transformation, 2 ms, corresponds to a few GPa pressure increase in the pressure chamber, so it is not possible to determine whether it this transformation time is due to the mechanism of the phase transformation or the pressure ramping. We have not been able to detect any intermediate phase during the transformation, which is not expected if Silcock mechanism is followed as suggested by orientation relations [3].

To sum up, the outputs of this experiment are:

- We have been able to record time-resolved XAS spectra at the ms timescale on Zr samples dynamically loaded in diamond anvil cell.
- We have detected the pressure-induced alpha-omega phase transformation in Zr around 12 GPa under quasi-hydrostatic loading, a pressure which is similar to the one observed with static loading for the same sample
- The transformation is rapid, less than 2 ms.


**References:**

[1] Singh *et al.*, J. Appl. Phys. 53, 1221 (1982)

[2] Ishimatsu *et al.*, J. Synchrotron Rad. 19, 768 (2012).

[3] Wenk *et al.*, Phys. Rev. Lett. 111, 195701, 2013


**Figure 1 : Main:** X-ray absorption spectra recorded in Zr dynamically loaded in a diamond anvil cell. Each thin curve is the average of 1 to 3 spectra recorded with a 0.5 ms exposure. The corresponding pressure, measured with time-resolved ruby luminescence technique, is indicated in the legend. The bold curves have been recorded statically, before and after the pressure jump. The different XAS spectra have been vertically shifted for clarity. **Inset:** pressure ramp for this experiment.