MLF Experimental Report

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課題番号 Project No. 2012A0036

実験課題名 Title of experiment
The crystal perfection and defect kinetics in the high temperature beta phase of Zr and Ti alloys: An in-situ study of primary extinction.

実験責任者名 Name of principal investigator
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装置名 Name of Instrument/(BL No.)
TAKUMI / BL19

実施日 Date of Experiment
2012/10/24 - 2012/10/27

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Axial symmetric, tensile specimens of were machined from solid bulk material, tapped at the end to fit the Takumi load frame. Three compositions were used in the following runs:

Zr-2.5Nb (mass %)	run # 10312, 10313
Ti (industrial grade II)	run # 10307, 10308, 10309, 10310, 10311
Ti-6Al-4V (mass %)	run # 10314

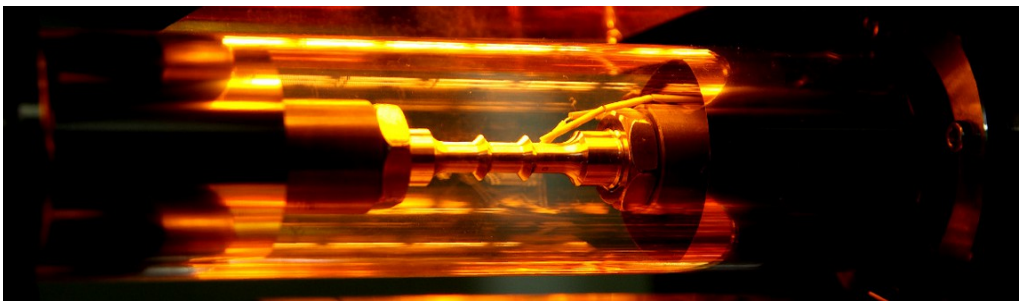

Figure1: *Sample as mounted in Takumi heated loadframe*

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）

Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

First, the instrument has been calibrated and short test runs (# 10303, 10304) were taken to chose for medium resolution configuration. A total of 8 runs on 5 different specimens were obtained on heating-loading cycles. Temperatures up to 1413 K and one or two cyclic plastic deformations of up to 1 mm span the parameter range. All experimental parameters were compiled into a master file as a function of real time,

starting from 0:00:00 o'clock of 2012/10/24, which is plotted in Figure 2 and allows to synchronize takumi, machine and loadframe data, each recorded on different computers.

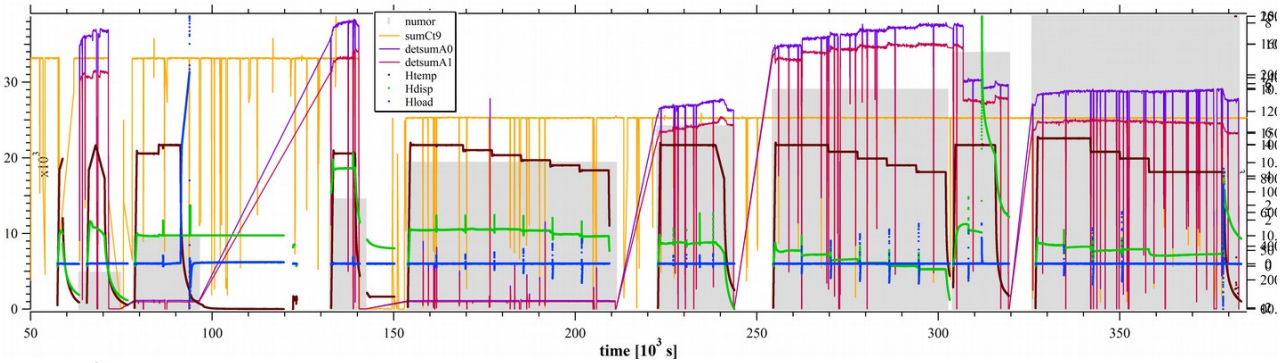

Figure 2: *Experimental parameters, such as file number, j-parc current, detector sum bench A and B, sample temperature, displacement and load as function of absolute time since start of the first day of experiment.*

Aim of the experiment was to observe the defect kinetics in the metallic samples at very high temperature, particularly upon distortion by plastic deformation. In a recent publication [S. Kabra et.al J. Appl. Phys (2013)] the authors report on extinction effects due to lattice perfection in Zr-2.5Nb, which established after recovery, i.e. annihilation of dislocations. The goal here was not only to record the temperature effect – furthermore, lattice distortions, i.e. dislocations were introduced into the system by plastic deformation.

A successful observation of such crystal recovery is seen in Figure 3, which shows the α-Zr 100, 002, β-Zr 110 and α-Zr 101reflections, evolving in time as the specimen is heated and processed. The huge line shifts before 700 s and after 11400 s are ascribed to thermal expansion upon heating and cooling as well as chemical effects [K. Yan et.al Adv. Eng. Mat. (2011)]. A transformation from α to β phase is expressed by the change of intensity between the reflections. Note the additional change of intensity of the β line (at 2.45 $Å^{-1}$, decreasing continuously while the temperature is held and increasing spontaneously around 4000 s and 8000 s, where plastic deformation (of 2 different magnitudes) was performed.

After α transformed to β phase, dislocations thermally annihilate, leading to more perfect crystallites, while intensity decreases due to primary extinction of neutron radiation. Upon plastic deformation, dislocations are induced into the crystallites, reducing extinction. Subsequently, the grain recover again and intensity weakens accordingly.

These diffraction observations are the first time recorded after plastic deformation and render the beamtime successful. Furthermore, similar effect has been seen on a second metal, namely Ti. More beamtime will be needed for consistent studies and to reveal physical parameters like activation energies. The effort needed for sample change was more than expected, and was the limiting factor for the in-situ studies. Some serious oxidation occurred, presumably due to long pumping lines, and it is suggested that a turbo-pump is installed close to the sample chamber. Higher maximum temperatures are desirable.

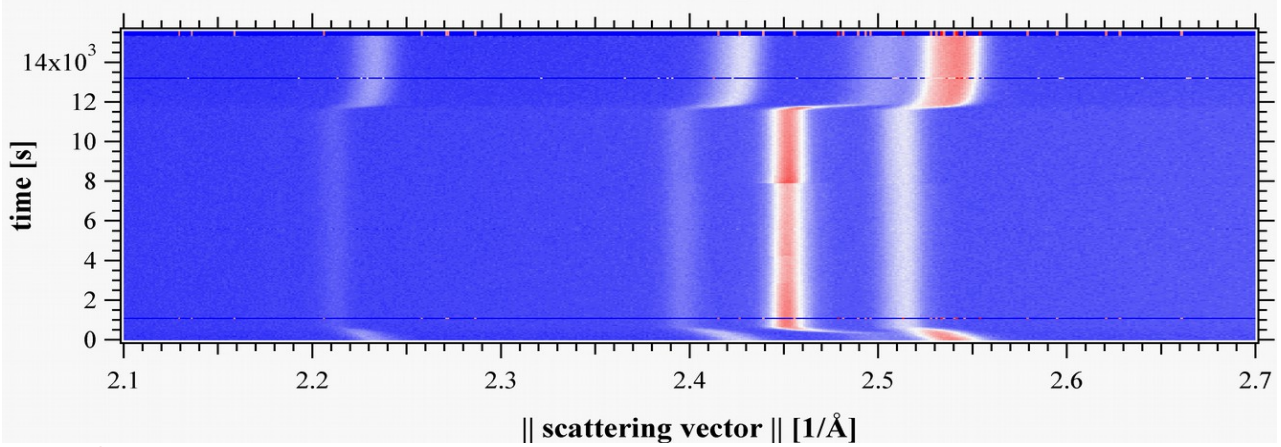

Figure 3: *Color-scaled powder diffraction patterns of Zr-2.5Nb as a function of momentum transfer and time. blue: low; white: medium; red: high intensity.*