ESRF	**Experiment title:** Time resolved studies of variant selection during phase transformation in hcp material	**Experiment number**: MA-270
Beam line: ID15b	**Date of experiment**: from: 27.06.2007 to: 02.07.2007	**Date of report**: 12-07-2009
Shifts: 12	**Local contact(s)**: John Elliott Daniels	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Mallikarjun Karadge, Dr. Michael Preuss, Joao Quinta da Fonseca*, Axel Steuwer, Javier Ernesto Romero Ospina*

School of Materials, The University of Manchester, UK

Nicholas Jones*, David Dye*, Richard Dashwood, Martine Jackson, Adam Stapelton*

Department of Materials, Imperial College, UK

Report:

Phase transformations in alloys often occur with specific orientation relationships between the precursor and transformed phases. This means that a crystallographic texture existing prior to transformation will lead to a specific texture in the transformed material. In titanium and zirconium alloys, there is a high temperature *bcc* phase, β, and a low temperature *hcp* phase, α, and the transformation from α to β and vice versa is governed by the so-called Burgers orientation relationship[1]. Because of crystal symmetry, there are 12 possible α orientations that can transform from a single parent β grain, i.e., there are 12 variants. If during transformation, individual orientations (or variants) occur/evolve more frequently than others, *variant selection* is said to have taken place, which in steels is usually associated with displacive phase transformation to martensite or bainite. However, in titanium and zirconium alloys variant selection is seen during diffusional phase transformation [2] and the effect can be exacerbated by application of stress during transformation [3]. Scientifically extremely interesting, variant selection in titanium and zirconium alloys is also of great importance for the aerospace and nuclear industry, since this phenomenon affects the texture development during thermomechanical processing of the material and consequently mechanical properties. To date, any variant selection studies have been carried out undertaking post-mortem studies using laboratory x-ray diffraction techniques or electron back scatter diffraction. However, since α to β phase transformation takes place over a large temperature range in the (α + β) phase region, it has to be expected that the degree of variant selection changes during cooling. In addition it is important to identify the root cause for any variant selection, which is believed to be found in the macro/micro stress state of the material. Hence it is important to characterise the evolution of variant selection and the stress/strain state simultaneously, which is only achieved by in-situ high energy synchrotron x-ray diffraction experiments.

During the beamtime granted, a number of in-situ loading experiments were carried out on different titanium and zirconium alloys using the electrothermal mechancial tester (ETMT) based on ID15. This enabled us to expose samples to specific thermal cycles while maintianing zero load or some load levels during phase trasnformation. The specimens were irradiated using a monochromatic high energy (~90keV) X-ray beam with a spot size of 250x250 μm. Debye-Scherrer rings were captured using the PIXIUM detector.

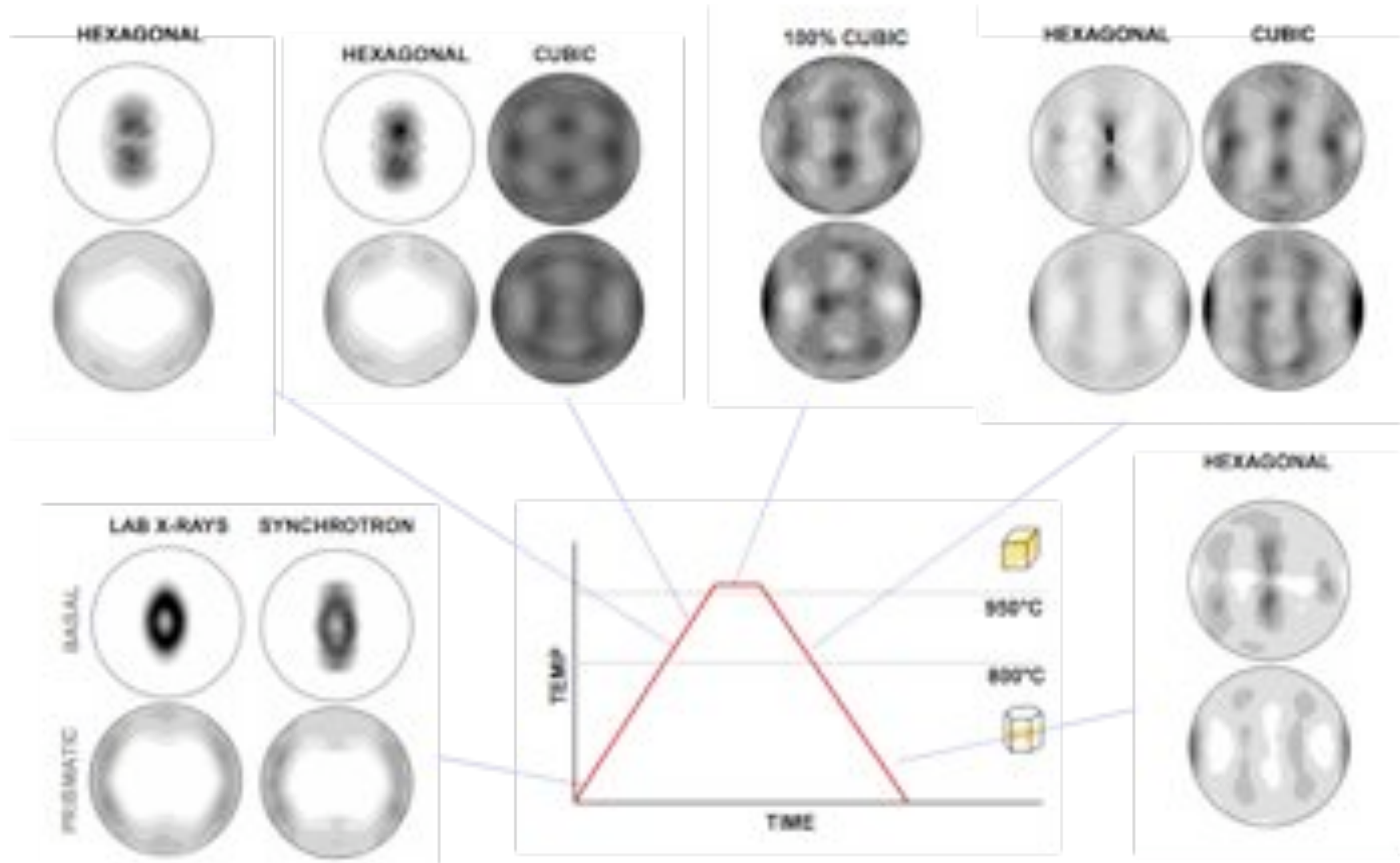

Figure 1. Texture evolution of the α and β texture during thermal exposure of Zircaloy-2 above the β-transus. The pole figures plotted are form the basal and (10-10) prosmatic plane in α and the (110) and (111) [4]plane in the β phase. It can be seen that in this specific case the strong rolling texture is first transformed into a recrystallisation texture before a complete β trasnformation and cool down has transformed the α texture more substantially.

It was possible to follow the texture evolution during heating and coooling zirconium and tianium alloys as shown in Figure 1. The experiments has provided new insight in variant selection under zero and low stress conditions. It was also found that in case the material was not completely heated above the β trasnsus complete texture memory effect was obserevd although the microstructure morphology had chnaged very significantly. This experiment was undertaken jointly by Manchester and Imperial. Since the set up for this experiment was ideal for a variaty of experiments about 4 PhD stduents benefitted from 4 days of beam time. As a result, 4 high quality journal publications have been produced from this experiment, which are listed below:

- J. Romero, M. Preuss, J. Quinta da Fonseca: Capturing the texture changes in a zirconium alloy during the allotropic phase transformation, Scripta Materialia, Volume 61, Issue 4, August 2009, Pages 399-402
- Adam M. Stapleton, Seema L. Raghunathan, Ioannis Bantounas, Howard J. Stone, Trevor C. Lindley, David Dye: Evolution of lattice strain in Ti–6Al–4V during tensile loading at room temperature, Acta Materialia, Volume 56, Issue 20, December 2008, Pages 6186-6196
- N.G. Jones, R.J. Dashwood, M. Jackson, D. Dye: β Phase decomposition in Ti–5Al–5Mo–5V–3Cr, Acta Materialia, Volume 57, Issue 13, August 2009, Pages 3830-3839
- J. Romero, M. Preuss, J. Quinta da Fonseca: Texture memory and variant selection during phase transformation of a zirconium alloy, accpted for publication in Acta Materialia, June 2009.

[1] C.S. Barrett and T.B. Massalski, Structure of metals (3rd ed.), McGraw-Hill, New York (1966).
[2] M. Humbert and N. Gey, Acta Mater. **51** (2003), p. 4783.
[3] N. Gey, E. Gautier, M. Humbert, A. Cerqueira, J.L. Bechade and P. Archambault, J. Nucl. Mater. **302** (2002), p. 175