ESRF	**Experiment title:** **Recrystallization and phase transformation kinetics of titanium-aluminides**	**Experiment number**: ME - 895
Beamline: ID15A	**Date of experiment**: from: 24.11.2004 to: 28.11.2004	**Date of report**: 31. 8. 2005
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Phase transitions and atomic rearrangement processes in polycrystalline substances play an important role in our daily life and are most important for the tailoring of modern materials. Multiphase alloys, such as titanium aluminides, bear distinguished mechanical properties depending on their thermo-mechanical treatment history and thus their microstructure. Much fundamental and industry-related research is undertaken to find the best process parameters. While metallurgical investigations are often obtained off-situ, little is known about the kinetics of the phase transition and the occurring atomic rearrangements. The importance of 2-D X-ray diffraction patterns and their relation to microscopic features has been demonstrated earlier [1]. Here we report on novel in-situ time resolved diffraction measurements which were taken at elevated temperatures. The transition from α-Ti_3Al to γ-TiAl grains has been followed in reciprocal space and is found to occur through an oriented rearrangement of atoms, which becomes observable only by rapid X-ray diffraction.

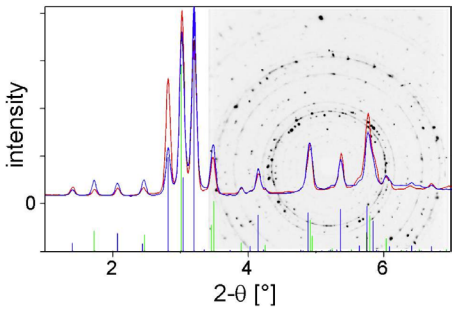

figure (1): *Even spiky patterns give reasonable results in Rietveld refinement.*

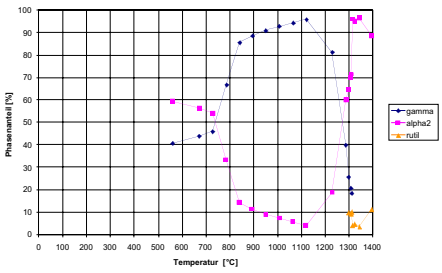

figure (2): *Phase composition upon heating a previously oil-quenched a-rich sample: First, the α-phase disappears at lower T in order to reappear at the α-transus temperature at 1300 C.*

The synchrotron measurements were obtained at the beamline ID15A at the European Synchrotron Radiation Facility ESRF where particularly high energy X-rays are available for bulk study experiments [2]. A fixed exit double monochromator defines the incident beam to the sample which scatters into Debye-Scherrer rings registered by a 2-D detector in the forward direction. We set up a commercial image intensifier tube coupled to the ESRF FReLoN CCD-camera in order to obtain data acquisition times between 0.3 s and 1 s. The 1-2 mm thick sample was surrounded by a cylindrical, vertically oriented furnace of 10 mm internal diameter. The temperature was measured by a thermocouple placed above the sample which turned out not to be very accurate. Thus temperature indications are obtained by complementary measurements (DSC) to which the observed data can be scaled. The furnace was mounted onto a vertical translation stage in order to be rapidly removed or repositioned over the sample, which resulted in fast heating and cooling rates. For the tiny sample as compared to industrial production

standards, however, the cooling rates in ambient air were often too fast in order to allow for nucleation and growth processes necessary for the massive transformation. Therefore we studied differently prepared samples mainly on rapid heating. Sometimes, the high-temperature glue was a problem when foaming at rapid heating rates and elevated temperatures above 1200°C occurred. This issue should be addressed for future experiments.

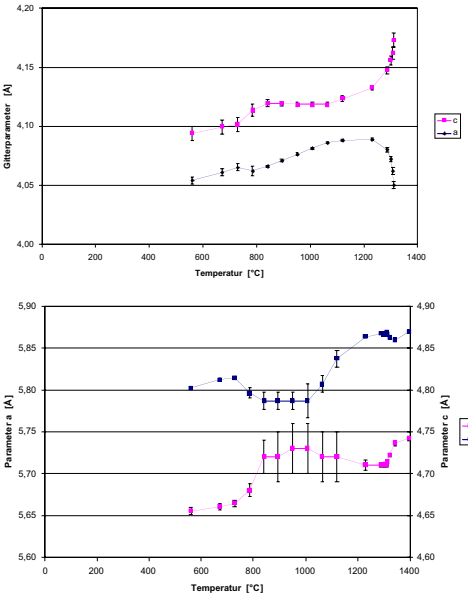

figure (3): *Evaluation of the lattice parameters in the γ- (top) and α-phase (bottom) as a function of temperature and off the thermodynamic equilibrium. Anomalies occur at the order-disorder transition around 900°C and chemical separation due to high diffusion takes place at 1300°C.*

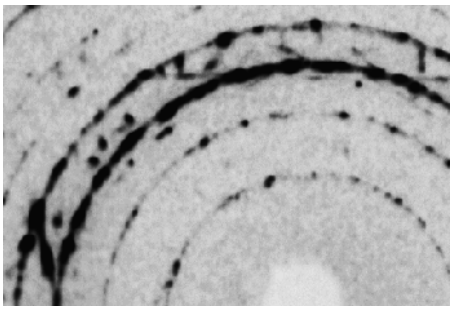

figure (4): Section of Debye-Scherrer rings indexed from the center γ-001, γ-110, α-200, γ-111/α-002, α-210, γ-002/200 in a snapshot during the transformation from the α- to the γ-phase. Streaks have been observed for a short time evolving from the α-210 rings to adjacent γ-111 or γ-002/202 rings. As a function of time, the γ-grains grow and increase the intensity of the spots while the α-intensity fades. The streaks can be interpreted as diffuse scattering from intermediate states during the phase transition while the atoms build a lattice gradient during rearrangement.

We have performed different combinations of temperature profiles and sample preparation. Data has been evaluated by making movies and 2-D representation as a function of time after azimuthal integration. Rietveld refinements allow to distinguish the phase compositions (figure (2)), the lattice parameters (figure (3)) and deviated results such as c/a ratios and thermal expansion coefficients. Anomalies and changes are observed at the α_2 _ α and (α+γ) _ α transitions, which not only depend on the temperature but also on time and thus on the kinetics of the system.

Diffuse diffraction streaks can be observed while the system transforms between the α- and γ- phase in the field of phase-coexistence. The movies demonstrate the time dependence and the flow of 'intensities' between spots of the α- and γ- reflections. Usually, the orientation between the grain of the new phase to the grain of the mother phase is described to be related by the so-called Blackburn-relation: The hexagonal base plane of the α-phase fits very well to the {111}-plane of the almost cubic γ-phase. The new data implies as well, that planes with greater lattice parameter (5%-10%) and orientation mismatch (~10°) transform through a preferred re-arrangement mechanism. It is a kind of transformation-recrystallization. The streaks represent the lattice parameter and orientation gradient in the front of the phase transformation where the atoms rearrange. Secondly, we anticipate that soft phonon-modes may be involved in the corresponding crystallographic directions which lead to thermal diffuse scattering. The streak at the phase-transformation will be a superposition of both effects. This observation is an experimental breakthrough in modelling transformation and recrystallization mechanisms which are also important in other situations and materials. It is being prepared for publication.

Altogether, the beamtime was experimental and successful revealing unexpected and novel aspects in the kinetics of phase transitions. The results are very rich in details and further investigations should be undertaken in order to tackle the different aspects. A higher resolution and linearity together with speed of the 2-D detector, such as the promising TRIXELL model, will be essential for more sophisticated studies.

Reference List

1. K.-D. Liss, A. Bartels, H. Clemens, S. Bystrzanowski, A. Stark, T. Buslaps, F.-P. Schimansky, R. Gerling, A. Schreyer: *"Recrystallization and phase transitions in a γ-TiAl based alloy as observed by ex- and in-situ high-energy X-ray diffraction",* Acta Materialia submitted.(2005)

2. K.-D. Liss, A. Bartels, A. Schreyer, H. Clemens: *"High energy X-rays: A tool for advanced bulk investigations in materials science and physics",* Textures and Microstructures **35-**3/4. 219-252 (2003)