In-situ X-ray diffraction for investigating the high temperature activation of a protective oxide layer on \( \gamma \)-TiAl

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The experimental aim of this study concerns the growing of a protective oxide layer on \( \gamma \)-TiAl base alloy substrate. \( \gamma \)-TiAl base alloy is used for high temperature applications (more than 750°C), such as gas turbines rotating in a gas flow. During the high temperature activation oxide layers are growing on the surface.

![Figure 1: a) 2D-detector with multichannel plate (MCP), MAXIM, DESY b) DHS 1100 heating unit, Anton-Par, with \( \gamma \)-TiAl specimen after the high temperature activation experiment](image)

The analyses were performed at the Beamline G3, MAXIM, HASYLAB at DESY in Hamburg (fig. 1a) \([1-3]\) equipped with the “DHS 1100 heating unit”, Anton-Par (fig. 1b). The space resolved 2D in-situ imaging of the surface area was performed using synchrotron X-ray radiation with the wavelengths \( \lambda = 0.2 \text{ nm} \) at constant psi and phi angles. A bundle of polycapillaries was arranged in front of a CCD-camera to enable position sensitive data acquisition. The \( \alpha \)-Al\(_2\)O\(_3\) \{211\} was measured. The detection system was wobbled during the measurement about \( \Delta 2\theta = 0.35^\circ \). The measuring time was fixed about 300s per image. The measured position sensitive intensity was adjusted to the I\(_{\text{Borrm}}\).

The object of the investigation was a polished \( \gamma \)-TiAl based alloy containing TiAl and TiAl\(_3\) phases on the surface (fig. 2a).

![Figure 2: oxide phases before a) and after b) the high temperature activation on the surface of the \( \gamma \)-TiAl specimen](image)

To start the high temperature activation the sample was heated up to 900°C and thus temperature was kept constant for 820 minutes. The heating and the cooling rate was 15 K/min at ambient air.

During the high temperature activation and oxidation a protective oxide layer of \( \alpha \)-Al\(_2\)O\(_3\), TiO\(_2\) and mixed oxide (Al\(_2\)Nb\(_2\)Ti\(_6\))O\(_2\) grew (figure 2b). During the experiment only \( \alpha \)-Al\(_2\)O\(_3\) was investigated.
Figure 3: Temperature distribution a), integral surface intensity a) and time dependent space resolved 2D X-ray distribution images b-d) of the $\alpha$-Al$_2$O$_3$ {211} reflection during the experiment. The spalling of the oxide layer (figure 3d) happened during the cooling down at $t = 779$ min d).

Before starting the heat treatment, only TiAl- and TiAl$_3$-phases are present on the surface (fig. 3b). During the heating the protective oxides of $\gamma$-Al$_2$O$_3$ grow. So the intensity at surface zone of the $\alpha$-Al$_2$O$_3$ depends on the time (fig. 3c). Caused by the residual stress formation during growing the lattice parameter is slightly shifted.

During the fast cooling down (fig. 3d-e) the protective surface layer is spalling and coarse grain starts to grow. The spalling oxides destroy the protective coating but on the surface oxides immediately start to grow again.

At the end of this experiment at ambient temperature only phases of $\alpha$-Al$_2$O$_3$, TiO$_2$ and mixed oxide (Al$_2$Nb$_2$Ti$_6$)O$_2$ are observed on the surface (fig. 2b), no reflections of TiAl and TiAl$_3$-phases were detected.

With these measurements we apply a recently newly developed method for in-situ space resolved X-ray phase distribution analyses. Additionally it is possible to visualize and animate the oxidation effects, inhomogeneous grain growth and the surface defects during the heating and the cooling period.

The next step will be the in-situ phase analysis and in-situ space resolved 2D X-ray diffraction during high temperature activation for different phases in one experiment ($\alpha$-Al$_2$O$_3$ and TiO$_2$) on polished surface and surface with riblet systems of protective oxides.

The authors would like to thank DFG for the financial support within the framework of SPP1299 ‘Adaptive surface for high temperature application’ [4]. Furthermore the authors would like to thank Prof. Ch. Genzel, Helmholtz-Zentrum Berlin, for providing the special heating unit and Dr. J. Donges, HASYLAB at DESY, Hamburg, for the experimental support.

References