

Experiment Report Form

The double page inside this form is to be filled in for each experiment at the Rossendorf Beamline (ROBL). This double-page report will be reduced to a one page, A4 format, to be published in the Bi-Annual Report of the beamline. The report may also be published on the Web-pages of the FZD. If necessary, you may ask for an appropriate delay between report submission and publication.

Should you wish to make more general comments on the experiment, enclose these on a separate sheet, and send both the Report and comments to the ROBL team.

Published papers

All users must give proper credit to ROBL staff members and the ESRF facilities used for achieving the results being published. Further, users are obliged to send to ROBL the complete reference and abstract of papers published in peer-reviewed media.


Deadlines for submission of Experimental Report

Reports shall be submitted not later than 6 month after the experiment.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the reference number of the proposal / experiment to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.
- bear in mind that the double-page report will be reduced to 71% of its original size, A4 format. A type-face such as "Times" or "Arial" , 14 points, with a 1.5 line spacing between lines for the text produces a report which can be read easily.

Note that requests for further beam time must always be accompanied by a report on previous measurements.

 ROBL-CRG	Experiment title: In-situ Evolution of Phase Composition and Internal Growth Stresses in Nanostructured Oxide Layers grown on Iron Aluminides	Experiment number: SI-1711
Beamline: BM 20	Date of experiment: from: 18 March 2008 to: 26 March 2008	Date of report: 24 November 2008
Shifts: 24	Local contact(s): Dr. Nicole Martha Jeutter	<i>Received at ROBL:</i>
Names and affiliations of applicants (* indicates experimentalists): Dr.-Ing. Haroldo Pinto*, Max-Planck-Institut für Eisenforschung, Düsseldorf Germany Dipl.-Ing. Pedro Paiva Brito*, Max-Planck-Institut für Eisenforschung, Düsseldorf Germany Prof. Dr. Anke Pyzalla, Helmholtz-Zentrum Berlin für Materialien und Energie, Berlin, Germany		

Report:

Iron aluminides are important materials for high temperature applications because of their ability to form an adherent α - Al_2O_3 scale on the metal surface. Critical to oxidation resistance of these alloys are growth stresses that develop within the scale during oxidation. Nevertheless, the origin of such stresses is not yet completely understood for many metal-oxide systems [Veal 2006].

The main objective of this project was to study the evolution of the growth stresses in oxide scales formed at low temperature (700°C). In this case a multi-layered oxide scale is formed, composed of α - Al_2O_3 and α - Fe_2O_3 , with an overall thickness of 80 to 100nm (figure 1).

In order to assess the influence of substrate microstructure and chemical composition on the oxide scale development, both single crystals and polycrystals of two Fe-Al alloys (ferritic Fe-15at.%Al and intermetallic Fe-26at.%Al) were used in the studies. The stresses in the growing

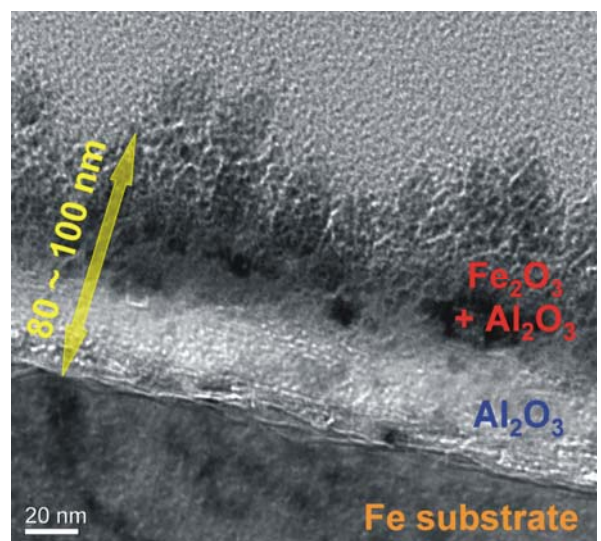


Fig. 1: TEM micrograph of the oxide scale grown on an Fe-15at.%Al substrate after 5h oxidation at 700°C .

in the scales were determined in-situ by applying the $\sin^2\Psi$ method with a grazing incidence angle of $0,3^\circ$ /Ma 2002/.

The evolution of growth stresses in the oxide layer formed on an Fe-15at.%Al single crystal is shown in figure 2. The stresses exhibit a transient behavior, in which initially elevated values gradually relax as oxidation progresses. In the initial stages of oxidation growth stress development was found to be governed by the epitaxy between the two main oxide phases present in the scale, due to reduced layer thickness /Panicaud 2006/. Because they share the same crystal structure $\alpha\text{-Al}_2\text{O}_3$ and $\alpha\text{-Fe}_2\text{O}_3$ exhibit parallel epitaxy relationships in the basal planes /Wang 2002/. Preliminary texture analyses have also indicated that the (104) planes of $\alpha\text{-Al}_2\text{O}_3$ and $\alpha\text{-Fe}_2\text{O}_3$ are also parallel to each other (figure 3). This is reflected in the development of the growth stresses, where the positive strain on the (104) plane of $\alpha\text{-Fe}_2\text{O}_3$ is balanced by compressive strains on the (104) plane of $\alpha\text{-Al}_2\text{O}_3$.

References:

1. B.W. Veal et al, Nat. Mater. 5 (2006) 349.
2. C.-H. Ma et al, Thin Solid Films 418 (2002) 73.
3. B. Panicaud et al, Appl. Surf. Sci. 252 (2006) 5700.
4. C.-M. Wang et al, Thin Solid Films 414 (2002) 31.

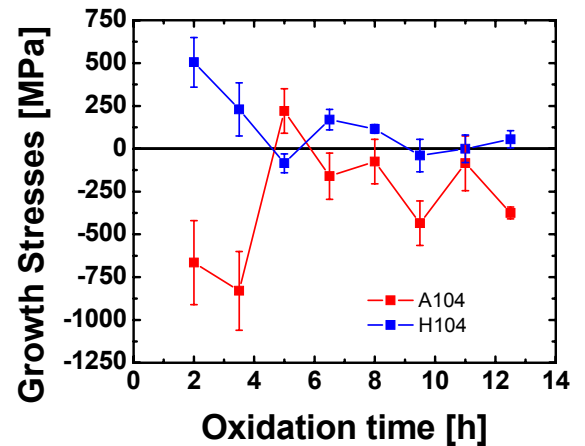


Fig. 2: Evolution of growth stresses in the oxide layer grown on an Fe-15at.%Al (100) single-crystal at 700°C.

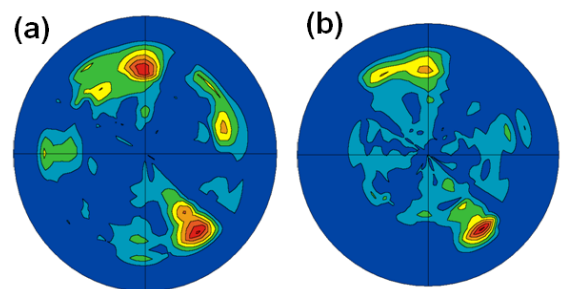


Fig. 3: Experimental pole figures of (a) $\alpha\text{-Al}_2\text{O}_3$ (104) and (b) $\alpha\text{-Fe}_2\text{O}_3$ (104).