MS20-P7 In-situ reduction of as-prepared γ-Iron Oxide Nanoparticles

Pelle G.R. Garbus[1], Jakob Ahlburg[1], Henrik L. Andersen[1], Lukas Keller[1], Mogens Christensen[1]

1. Interdisciplinary Nanoscience Center, Aarhus university, Denmark

email: garbus@inano.au.dk

Magnetic materials are a hot topic among energy-materials and they find applications in nearly all everyday consumer electronics. Advances in magnetic performance have in particular been made for thin film and nanosized particles, because the magnetic properties are strongly related to the size. Bulk iron is relatively unreactive, however iron on the form of nanoparticles are highly reactive due to the enlarged surface area and the oxidation potential of iron. Iron oxides are cheap and unreactive precursors for the production of nanosized iron particles. Understanding the mechanisms behind the structural development [1, 2] adds to the fundamental understanding of materials' formation and can lead to new synthesis pathways. In this study, iron oxide (γ-Fe_2O_3) particles were heated to 400°C under a flow of H_2/Ar mixture, while the process was followed by *in situ* synchrotron powder X-ray diffraction measurement. The as-prepared maghemite nanoparticles were synthesized by the continuous decomposition of solutes in supercritical hydrothermal flow synthesis [3, 4]. The reagent used was ferric ammonium citrate ($C_6H_8O_7$•xFe(III)•yNH_3) that under hydrothermal flow synthesis decomposes into the γ-iron oxide Fe_2O_3. The reduction of maghemite to body centered cubic (BCC) iron does not go through a detectable intermediate state.

1. Jensen, K.M., et al., *Mechanisms for iron oxide formation under hydrothermal conditions: an in situ total scattering study.* ACS nano, 2014. **8**(10): p. 10704-10714.

2. Andersen, H.L., et al., *Size and Size Distribution Control of γ-Fe_2O_3 Nanocrystallites: An in Situ Study.* Crystal Growth & Design, 2014. **14**(3): p. 1307-1313.

3. Bondesgaard, M., et al., *Guide to by-products formed in organic solvents under solvothermal conditions.* The Journal of Supercritical Fluids, 2016. **113**: p. 166-197.

4. Bremholm, M., M. Felicissimo, and B.B. Iversen, *Time-Resolved In Situ Synchrotron X-ray Study and Large-Scale Production of Magnetite Nanoparticles in Supercritical Water.* Angewandte Chemie, 2009. **121**(26): p. 4882-4885.

Keywords: In situ PXRD, reduction, maghemite, iron oxides, magnetic materials

MS21 Structural disorder and materials' properties at ambient and non-ambient conditions

Chairs: Dmitry Chernyshov, Vaughan Gavin

MS21-P1 HRXRD analysis of bonded Si / Si interface

Zoltán Balogh-Michels[1], Zweiacker Kai[1], Zhang Yucheng[2], Jung Arik[2,3], Flötgen Christian[4], Chahine Gilbert[5], Dommann Alex[1], Erni Rolf[2], von Känel Hans[2,3], Neels Antonia[1]

1. Center of X-Ray Analytics, Empa, Swiss Federal Laboratories for Materials Science and Technology, 8600 Dübendorf, Switzerland
2. Electron Microscopy Center, Empa, Swiss Federal Laboratories for Materials Science and Technology, 8600 Dübendorf, Switzerland
3. Laboratory for Solid State Physics, ETH-Zürich, Schafmattstrasse 16, 8093 Zürich, Switzerland
4. EV Group, 4782 St. Florian/Inn, Austria
5. European Synchrothron, ESRF, Grenoble 38043, France

email: zoltan.balogh@empa.ch

Stress and strain can not only influence the structural behavior of the materials but can significantly alter their functional properties. The need for local, nanoscale characterization of stress levels and its correlation with other material properties increases rapidly. Scanning X-ray microscopy using synchrotron radiation is an emergent technique which can deliver fast, conclusive results with *submicrometer real space* resolution [1,2]. On the other hand the technique is limited in its *reciprocal space* resolution by the pixel size of 2D detectors. This is especially important if high quality single crystals have to be characterized. Modern laboratory instruments therefore offer the complementary capability owing to their higher reciprocal space resolution.

In this study we investigated the stress distribution in Si wafer pairs which were covalently bonded at room temperature [3]. The wafer bonds were analyzed by the "nanodiffraction" ID01 beamline at ESRF (F) [1] as well as by a Bruker D8 Davinci HRXRD instrument at EMPA (CH). Transmission electron microscopy (TEM) was used for morphological analysis. The specific wafer bond shown in Fig. 1 exhibited bulk bond strength, but contained a ~ 3 nm thick amorphous interfacial layer in their as-prepared form. After high temperature annealing a network of dislocations emerged to compensate for rotation and tilt of the two wafers.

The built-in stress at the interface caused some long range changes in the diffraction patterns, which can easily be distinguished by lower spatial resolution laboratory scale devices. The evaluation of the rocking curve FWHM going through the bonding interface cross section (X-ray beam size 50mm) shows the overall silicon crystal