Report on experiment SI 1072 : Oxidation of single crystalline surfaces of iron whiskers

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As described in the proposal of the experiment, the surface of a Fe whisker was considered to be suitable for fundamental oxidation studies since it was expected that its structural quality would be superior to that of conventional Fe single crystals.
A "large" whisker (0.5x0.5x15 mm^3) provided by Dr. D. Sander (MPI Halle) was employed in the experiments. It was mounted in a UHV sample holder and heated by conduction with a ceramic heater. The sample was installed in the UHV-high pressure chamber of ID3.
The 0.25 mm^2 surface was illuminated by a x-ray beam of approximate dimensions 100 μm H x 20 μm V . As the angle of incidence was set to 1 °, only 9 μm of beam were intercepted by the sample surface. This had as consequence that diffraction from the side faces was also present superimposed to that from the top surface.
Conventional methods were employed to try to clean the surface namely Ar sputtering followed by annealing. They revealed inefficient since FeO on the top and side surfaces was always present as reveled from powder diffraction scans. After many unsuccessful trials, hydrogen treatments at elevated temperatures (~800 K) were found to be the best method to clean the surface. As a result, clean surfaces with good crystalline quality were achieved.

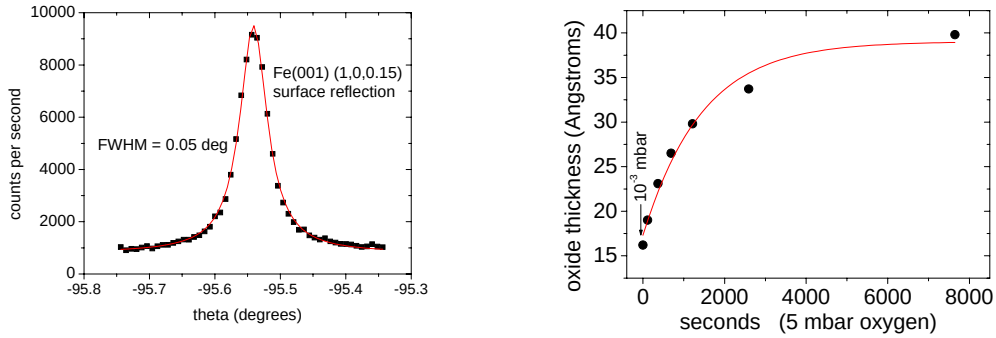

Fig 1 (left) : Surface reflection (1,0,0.15) from the top surface of the Fe whisker after cleaning. The FWHM of 0.05 degrees is about 6 times smaller than that obtained in a conventional Fe (001) crystal of standard dimensions.
(right): Oxidation of the Fe (100) surface under 5 mbar of oxygen at room temperature.

As observed in Fig 1 (left) close to an antiphase position in a CTR the intensity is 9kc/s and the width corresponds to ordered areas of 2.866Å/πx0.00084 rlu ≅ 10^3Å. The relatively high peak intensity and low background would allow to measure rather easily surfaces of lateral dimensions of about 0.17 mm which should be even better ordered.

Figure 1 (right) shows the thickness of the oxide as a function of time when the surface was exposed to 5 mbar of O_2 at room temperature. The thickness was determined from the spacing of the fringes of the specular reflection caused by the lower density of the oxide film compared to that of the metal. The maximum thickness achieved is 40 Å

which is more than two times larger that the maximum reported thickness for oxidation under HV conditions (10^{-6} mbar of O_2) . This poses fundamental questions to the theory of metal oxidation currently accepted.
We also investigated the structure of the oxide and we found that it consists in an epitaxial magnetite film.