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Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

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 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.

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	Experiment title: EXAFS investigation of austenitic alloys nitrided at low temperature	**Experiment number**: 20-01-686
Beamline:	**Date of experiment**: from: 02/09/2009 to: 04/09/2009	**Date of report**: 18/05/2010
Shifts:	**Local contact(s)**: Andreas Scheinost	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Chemical environment of the principal constituents (Fe, Ni, Cr) of the austenitic stainless steel (ASS) AISI 316L in the S phase has been investigated by means of x-ray absorption near edge structure (XANES) and extended x-ray absorption fine structure (EXAFS). The S phase has been produced by plasma nitriding at 400°C for 30h. This results in the nitrided layer thickness of ~20 μm and nitrogen near-surface atomic ratio of ~27 at.%. In order to get some insights into the chemical environment of Fe, Ni, Cr, the XANES and EXAFS spectra of virgin ASS and ASS plasma nitrided at 550°C for 30h have been recorded. The former serves as a reference for Fe, Ni, Cr in fcc metallic state. Nitriding at 550°C induces the precipitation of CrN which serves as a reference for Cr in CrN state.

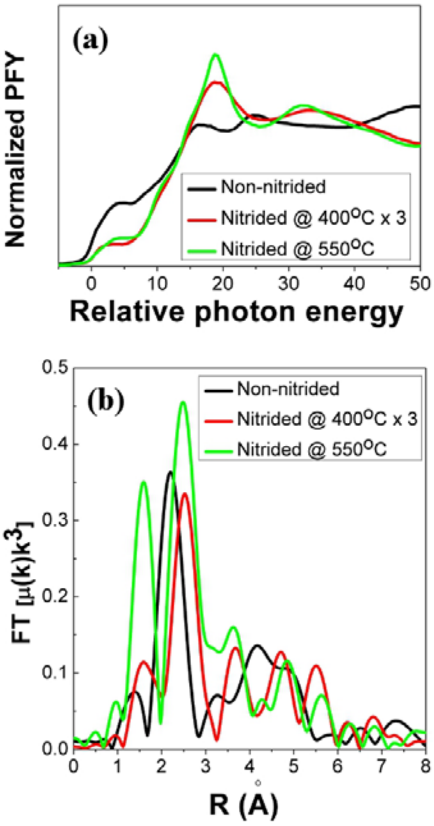

Figure 1. Chemical environment of Cr in metallic (non-nitrided), CrN (nitrided at 550°C) and S phase (nitrided at 400°C) states: XANES partial fluorescence yield (PFY) (a) and Fourier Transform (FT) of EXAFS oscillations (b).

Figure 1 summarizes the XANES and EXAFS data on chromium. The XANES and k^3 weighted Fourier transform of the EXAFS spectrum (FT|$\chi(k)k^3$|) of the virgin ASS sample are characteristic for the *fcc* structure. There are four strong amplitude peaks in FT$|\chi(k)k^3|$ between 1.5 and 6 Å whose distance *R* are ~2.2, 3.2, 4.2 and 5 Å, respectively. The peaks correspond to metallic neighbors in different coordination shells. The shape of the K-edges for all elements is similar (not shown) which suggests that the chemical environments and geometry are the same for Ni, Fe and Cr. Cr XANES of the ASS nitrided at 550°C has a well-defined edge feature (~5 eV), main peak (~18.8 eV), and a second peak (~32 eV) which almost identical to CrN XANES reported in the literature. The edge is shifted by 0.44 eV as compared to non-nitrided sample indicating that Cr is in the oxidation state similar to Cr^{3+}. This corresponds to the formation of the CrN. FT$|\chi(k)k^3|$ shows additional coordination shell closer than any Me-Me which corresponds to N atoms. For the sample nitrided at 400°C, the Cr XANES is very similar to that of the sample nitrided at 550°C. Also the edge shift is close to that of the CrN reference sample. The peaks above the absorption edge are less intense while their positions are slightly shifted to the higher energies. The latter is attributed to slightly smaller lattice parameter, while the smoother shape can be attributed to a lower order of crystallinity. FT$|\chi(k)k^3|$ shows very similar coordination shell structure as that of CrN. The results point out that the Cr in the S phase is in the state similar to that of CrN. Similar analysis of other elements (not shown here) shows that Fe in the S phase is in the environment close to Fe_4N while Ni is in metallic disordered environment.

The results above strongly indicate that the interstitial nitrogen incorporation into ASS at 400°C results in the local decomposition of otherwise homogeneous solid solution of Fe-Ni-Cr, each principal element showing different local environment. This points out that the diffusion of the matrix atoms takes place even at the temperatures as low as 400°C. The fact that no different phases are observed by x-ray diffraction points out that such zones must be small and coherent with the matrix. Such composite structure explains the mechanical properties as well as ferromagnetism of the S phase reported in the literature. These findings show that reactive interstitial diffusion can be used to induce the local decomposition which shows a unique set of properties. These results make a part of the PhD thesis of A. Martinavičius (under preparation at the moment). The preparation of a publication is planned in the nearest future.