

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.



	Experiment title: Structural characterisation of surface modified materials by using REFLEXAFS (III): Nitrides	Experiment number: ME-737
Beamline: BM29	Date of experiment: <i>(Long term project)</i> from: Dec-2003 to: Jun-2005	Date of report: Feb-2007 Received at ESRF:
Shifts: 72	Local contact(s): Silvia Ramos; Gianluca Ciatto	
Names and affiliations of applicants (* indicates experimentalists): <div><div>Adela Muñoz Páez; Víctor López-Flores</div><div>Stuart Ansell; Daniel T. Bowron</div><div>Sofía Díaz-Moreno</div><div>Silvia Ramos</div></div> <div><div><i>Institute of Materials Science of Seville.</i></div><div><i>ISIS, Rutherford Appleton Laboratory.</i></div><div><i>Diamond Light Source, Ltd.</i></div><div><i>University of Birmingham.</i></div></div>		

Report:

One of the most important surface modification treatment in industry is the nitriding. In this process, the surface of an steel or an alloy is hardened by a surface layer of nitrides made from the transition metals that the steel or alloy contains, which normally are Ti, V, Cr or Mo among others. As a first approach, a study of bulk nitrides was made by transmission EXAFS.

Ternary Ti-V nitrides were synthesized by two methods. The first sample, TiVN-Carb, was prepared from carbothermal reduction in nitrogen atmosphere of TiO_2 and V_2O_5 oxides, which were previously milled for 1 h in a planetary mill. The second sample, TiVN-Mill, was prepared by milling pure Ti and V metals in nitrogen atmosphere for 10 h.

The XRD spectra at Fig. 1 show well defined peaks at angles intermediate between those of TiN and VN for TiVN-Carb sample. In contrast, the peaks of TiVN-Mill are not so well defined, thus indicating a sample rather amorphous.

The EXAFS spectra were recorded in transmission mode. Both Ti-K and V-K absorption edges were recorded in the same spectrum. In the general spectrum (Fig. 2), a difference between the spectra of both samples can be seen: TiVN-Mill has a quicker decay of the EXAFS oscillations than TiVN-Carb. This may be due to a higher milling time that may have caused a higher amorphization of the structure, reducing the local order around the absorbing atoms.

The spectra were simulated and fit by the FEFF 6 and FEFFIT 2.54 codes. The main variables were a single variable changing the lattice parameter, that allowed the coherent variation of the coordination distances, and the Debye-Waller factors.

The results for TiVN-Carb show that the local environments of both Ti and V maintain the Debye-Waller factors relatively low, thus indicating a relatively ordered sample in the short range. However, a different lattice parameter is obtained in each edge. Then, the Ti and V nitrides may be not forming an unique perfect TiVN₂ phase.

The results for TiVN-Mill show several things: first, there is some remaining Ti and V metal in the samples. However, Ti metal local environment was fitted with a *fcc* structure. Then, Ti metal seems to have undergone a polymorphic transformation from the stable *hcp* to *fcc* induced by the intensive milling. This has been previously described in [1], and the influence of the newly synthesized TiN-*fcc* structure may have also enhanced this effect. On the other hand, V metal keeps its *bcc* structure. Second, the Debye-Waller factors are significantly higher than those of TiVN-Carb. This is expected due to the intensive milling that might have distorted the crystal structure. Finally, since both environments at Ti and V-K absorption edges show very similar structural parameters, including the lattice parameter, it is concluded that this sample contains an unique TiVN₂ ternary nitride.

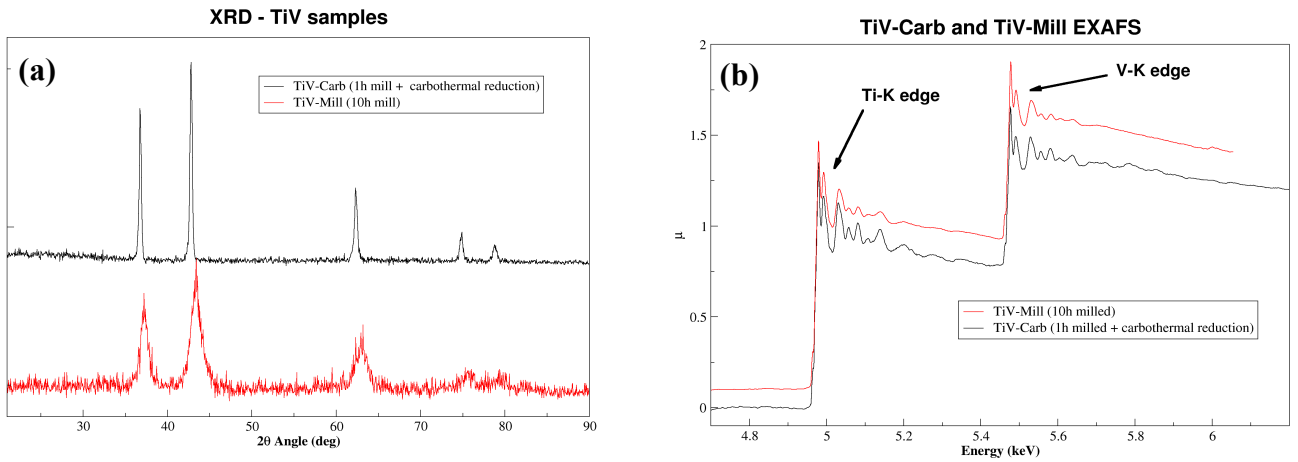


Fig. 1: (a) XRD spectra of both samples. (b) General absorption spectrum of both samples.

[1] I. Manna, P.P. Chattopadhyay, P. Nandi, F. Banhart, and H. J Fecht. *J. App. Phys.* **93** 1520 (2003)