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 via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can 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.

ESRF	**Experiment title:** Phonon confinement in Fe nanowires	**Experiment number**: HC-1657
Beamline: ID18	**Date of experiment**: from: 26Aug 2014 to: 02 Sep 2014	**Date of report**: 27/02/2015
Shifts: 18	**Local contact(s)**: Dr. Daniel geza Merkel (email: dmerkel@esrf.fr)	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

D.Bessas[1,*], J. G. Fernandez[2,3,*], V. V. Martinez[2,*] , V. M. De la Prida[2], K. Nielsch [3]

[1] European Synchrotron Radiation Facility, FR 38043, Grenoble, France.

[2] Universidad de Oviedo, Departmento de Fisica, Facultad de Ciencias, ES-33007, Spain

[3] Universitat Hamburg, Institute of Applied Physics and MRC, DE-20355, Germany

Report:

In the allocated beamtime we investigated the influence of size effects on the phonon properties. Our aim was to specifically concentrate on the Debye level of low dimensional materials, such as Fe nanowires grown in amorphous alumina templates using a novel electrodeposition technique. The used alumina templates had controlled pore diameter which provided nanowires with homogeneous diameter.

We measured nuclear forward and inelastic scattering spectra at ID18/ESRF using a high resolution monochromator with instrumental resolution of 0.5 meV. The measurements were carried out in two directions, *i.e.*, with the incident beam parallel and perpendicular to the nanowire axis. This was done in order to identify potential anisotropy in the vibrational properties of our systems. However, as it is expected from the isotropic crystallographic unit cell of α-Fe anisotropicity was not observed.

The data acquisition procedure was smooth and the quality of the obtained data was exceptional. The measured raw nuclear forward scattering is presented in Fig. 1. The obtained density of phonon states is presented in Fig. 2.

The nuclear forward scattering spectra are similar and independent from the nanowire diameter. Only the sample with 40 nm diameter wires appears to have different beating than all the other samples. After a detailed analysis of the raw data we could identify impurity phases which had hyperfine parameters different than that of pure α-Fe not only for the 40 nm diameter wires but also for the other nanowires.

The nuclear forward scattering data were corroborated with Mossbauer data. The Mossbauer data were obtained in order to resolve potential impurity phases which mainly contribute in the unresolved time, before 10 ns, in the nuclear forward spectra. A model was developed with which both nuclear forward spectra and Mossbauer data were fitted simultaneously. We were able finally to obtain a quantified analysis of the impurity phases.

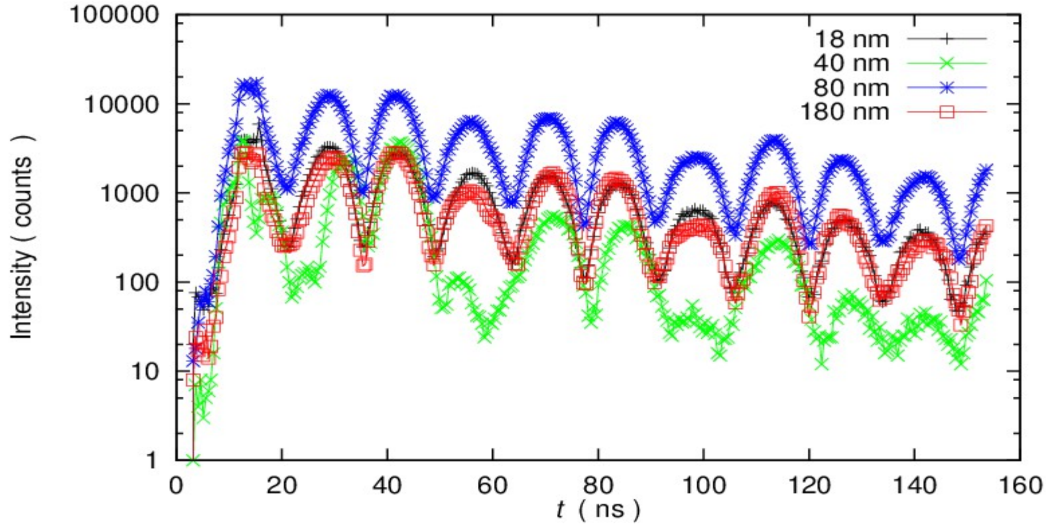

Fig. 1: The nuclear forward scattering spectra obtained on 20, 40, 80 and 180 nm diameter Fe nanowires with $k \perp$NW.

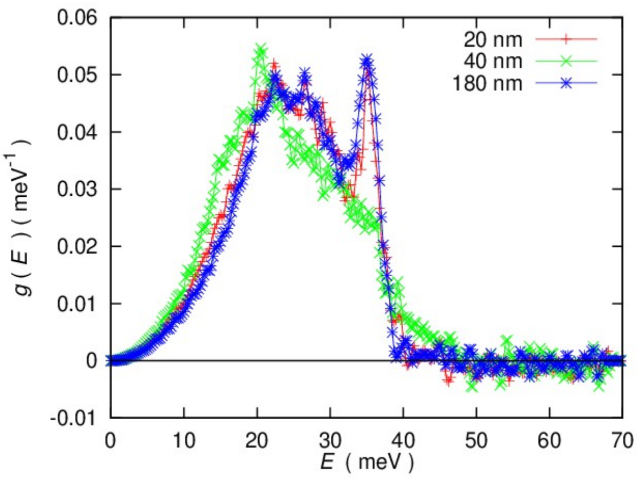

Fig. 2: Fe specific DOS measured in 20, 40 and 180 nm diameter Fe nanowires with $k \perp$NW

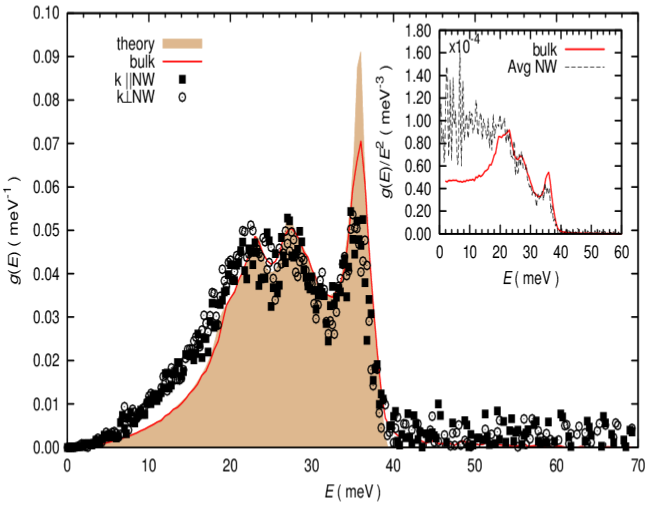

Fig. 3: Fe specific DOS measured in two geometries on 20 nm diameter Fe NW, with k ||NW and with $k \perp$NW, and comparison to similar measurements on bulk single crystals and theoretical calculations. Inset: comparison of the Debye level between bulk sample and NW.

The density of phonon states seem to have no strong diameter dependence in the examined diameter range, between 20 and 180 nm, except for the 40 nm samples where significant impurity phases were identified. However, the obtained density of phonon states of Fe nanowires is different than the analogous measured in bulk samples as well as the one obtained from first principle theoretical calculations, see Fig. 3. Nevertheless clear phonon peaks at energies higher than the cut-off energies for α-Fe, *i.e.*, at ~40 meV, are not observed up to 70 meV, which indicate that foreign Fe phases should not be present in the samples. However, this observation contradicts the nuclear forward scattering spectra obtained on the same samples. Combining the results obtained in this study with further investigations (Mossbauer spectroscopy, diffraction using synchrotron radiation, and SEM, TEM) we can conclude that, although phonon confinement in Fe nanowires with diameter down to 20 nm may not play the major role, there is definitely a change in the elastic properties which is probably related with the sample preparation procedure and results in a dramatic change in the thermal properties by a factor of 8.