EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Nucleation and growth of MOFs (subexperiment: local structure of V and Nb additives in TiFe alloys)	**Experiment number**: A31-1 173
Beamline: BM31	**Date of experiment**: from: 11.06 to: 14.06	**Date of report**: 12.10.2022
Shifts:	**Local contact(s)**: Dragos Stoyan	*Received at ESRF:*

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Report:

The goal of this measurement was to identify local structure around Nb and V in TiFe alloys. TiFe alloys are an attractive compound for hydrogen storage, and additives were shown to improve various hydrogen storage properties of these alloys. Different additives can occupy Ti or Fe cites, or both. Property modification depends on which cites are substituted and whether the substition is stable upon cycling in hydrogen. We have prepared samples containing only Nb, only V and both Nb-V additives. In this experiment were had little time to measure these compounds. The synchrotron XRD data of most of the samples were also obtained at one of the previous sessions.

Measurements at Nb k-edge

The Ti-Fe-Nb-V samples with different concentrations were measured. Figure 1 shows data quality for one of the samples. 2-3 scans were merged to obtain the spectrum:

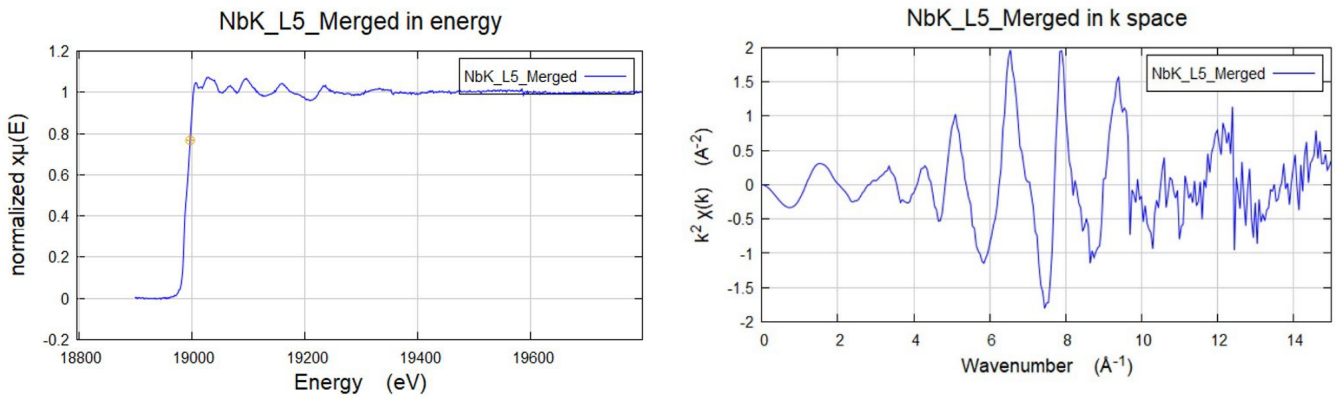

Figure 1. Representative data quality for the measurement at Nb k-edge of Ti-Fe-V-Nb alloys with <1 at.% Nb concentration.

Figure 2 shows the comparison of the series of Ti-Fe-Nb-V with various concentration of Nb and V and with the Nb foil. The figure clearly shows that the Nb atoms are diluted in the TiFe matrix. This conclusion is made due to the difference in the local environement of Nb in metal and in the alloy rulling out the formation of Nb metallic clusters / inclusions. This conclusion is also confirmed by the sychrotron data of the compounds that did not find metallic Nb in the samples (Figure 3). The figure also shows a variation in Nb local structure in different compounds.

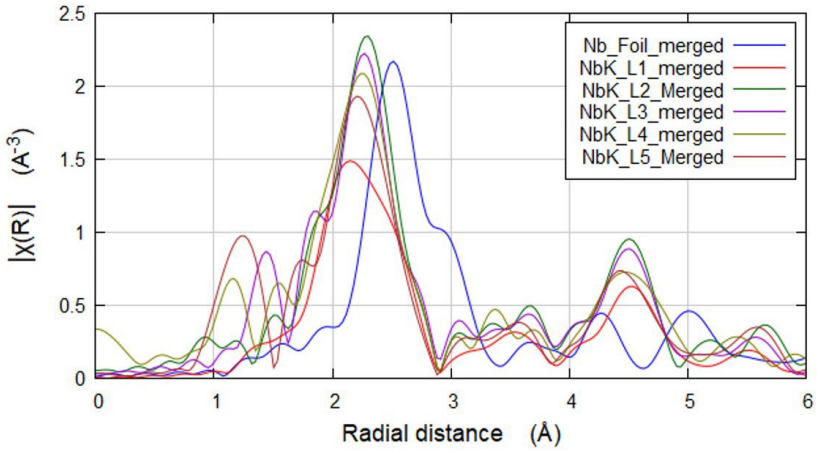

Figure 2. Nb in the TiFe alloys with various concentrations

This is a preliminary analysis. The data reduction was improved for the EXAFS fitting in order to reduce the abnomalous peak at 1.2 Å.
The fitting of EXAFS is being carried out in correlation with Rietveld analysis of the synchrotron data of the samples. We use various supercells and their combinations to solve the problem of partial occupancies. A good agreement between the occupancies from Rietveld refinement and the EXAFS fitting is obtained.

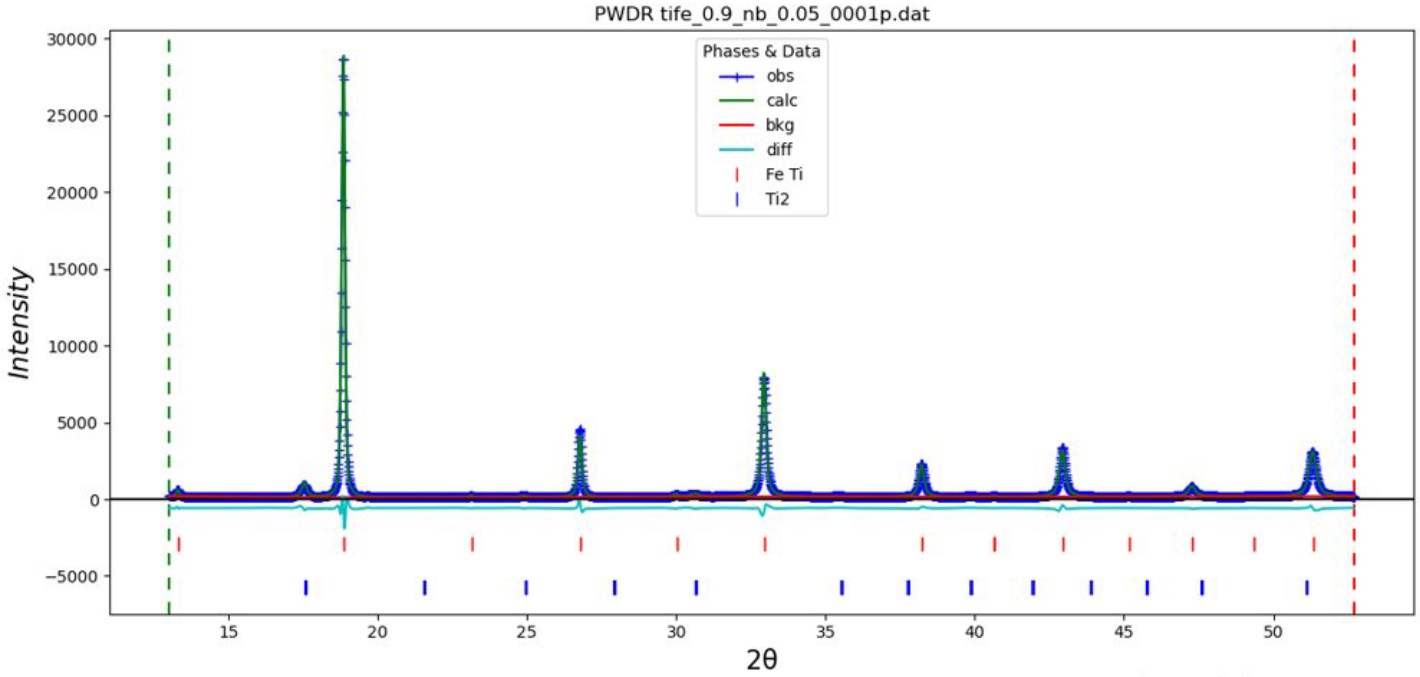

Figure 3. Rietveld refinement of synchrotron PXRD data of a Ti-Fe-Nb alloy.

Measurements at V k-edge

These measurements were not very successful as such a low energy in combination with low concentration required longer measurement time that we did not have in this experimental session. We will consider this in the future applications. Figure 4 below shows the measurements at V k-edge

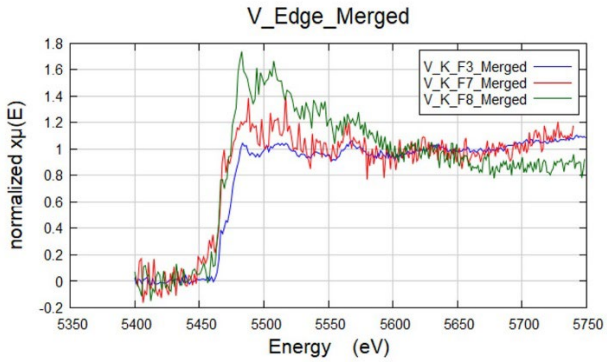

Figure 4. Ti-Fe-V-Nb alloys measured at V edge.