ESRF	**Experiment title: Study of the microstructure of INVAR FeCr alloys by high resolution powder diffraction.**	**Experiment number**: HS3047
Beamline: BM25	**Date of experiment**: from: 12/4/06 to: 14/4/06	**Date of report**: 10/6/08
Shifts: 6	**Local contact(s)**: German R. Castro	*Received at ESRF:*

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We investigated the microstructure of INVAR ball milled FeCr alloys by high resolution powder diffraction (HRPD) and X-ray Absorption spectroscopy (XAS) on the beamline BM25. The main objectives of this study, as stated in the proposal HS3047, were to probe purity of the mechanically alloyed FeCr alloys and to get an idea of their crystallinity and microstructure.

First of all, x-ray powder diffraction patterns were taken at room temperature for the $Fe_{30}Cr_{70}$ composition investigated. Due to the limited instrumental resolution and to the very similar lattice parameters for α-Fe and α-Cr body-centered cubic crystal structures ($a_{\alpha\text{-Fe}}$ = 2.886 Å, $a_{\alpha\text{-Cr}}$ = 2.8849 Å) HRPD measurements were not sufficiently succesful in determining the single phase or double phase character of the alloys.

However, the high versatility of the beamline BM25 allowed to change the setup very quickly and perform XAS measurements at both the Cr K edge and the Fe K edge at room temperature. Pellets were prepared by diluting the FeCr powders with BN. Thickness and homogeneity of the samples were optimized to obtain the best signal-to-noise ratio, giving a total absorption edge jump of 2. Calibration of the edge energy were performed with a Cr foil. Our spectra were all recorded in the transmission geometry.

Results of the XAS measurements are presented in Figure 1. From the Extended X-ray Absorption Fine Structure (EXAFS) region that extends from 50-1000 eV, a decrease in the

amplitude is observed (see Figure 2). A comparison with the EXAFS signal of a Cr foil crystalline standard shows a similar structure (bcc). A preliminary analysis of the EXAFS spectrum corresponding to the as-milled sample seems to suggest a decrease in the coordination number. However, different effects can affect the level of the EXAFS signal like the Debye-Waller factor, accouting for thermal and structural disorder or the photoelectron mean free path. New experiments performed at low temperature will be a key to discern the effects of thermal disorder and the structural disorder.

The analysis is in progress and a preliminary work has been presented at the University of Porto, during the Ninth-International Workshop on Non-Crystalline Solids [P-69 "Structural and magnetic evolution of mechanically alloyed $Fe_{30}Cr_{70}$ studied by neutron thermodiffractometry and x-ray absorption spectroscopy", A. Fernandez-Martinez, D. Martinez-Blanco, M. J. Perez, G. J. Cuello, G. R. Castro, J. A. Blanco, P. Gorria].

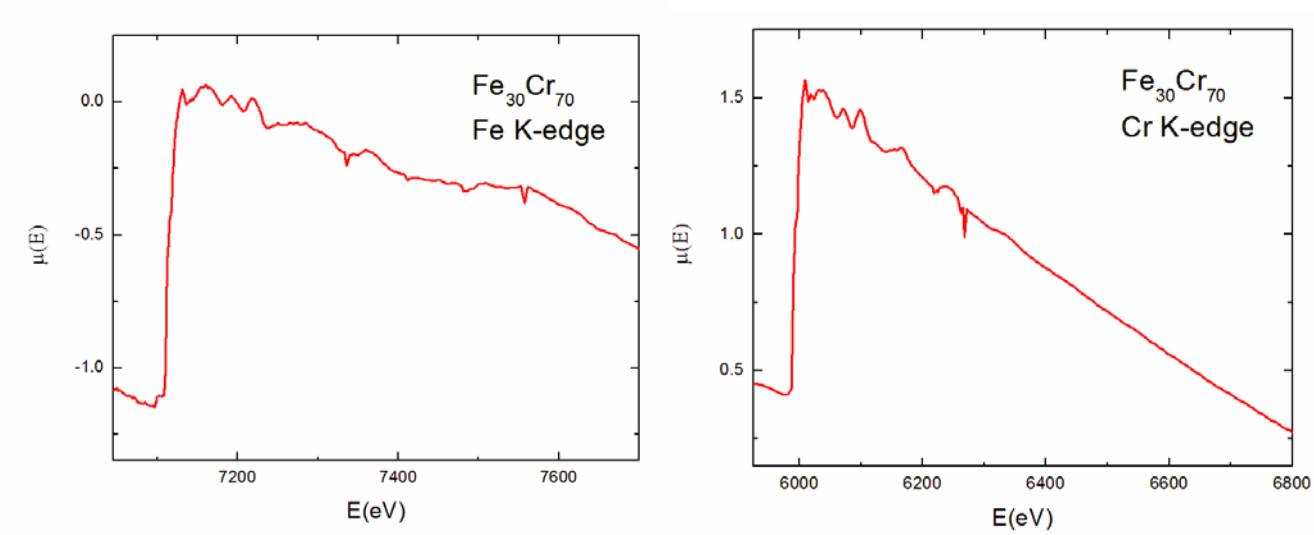

Figure 1. XAS spectra measured at the Fe (left) and at the Cr (right) K edges in ball milled $Fe_{30}Cr_{70}$ sample.

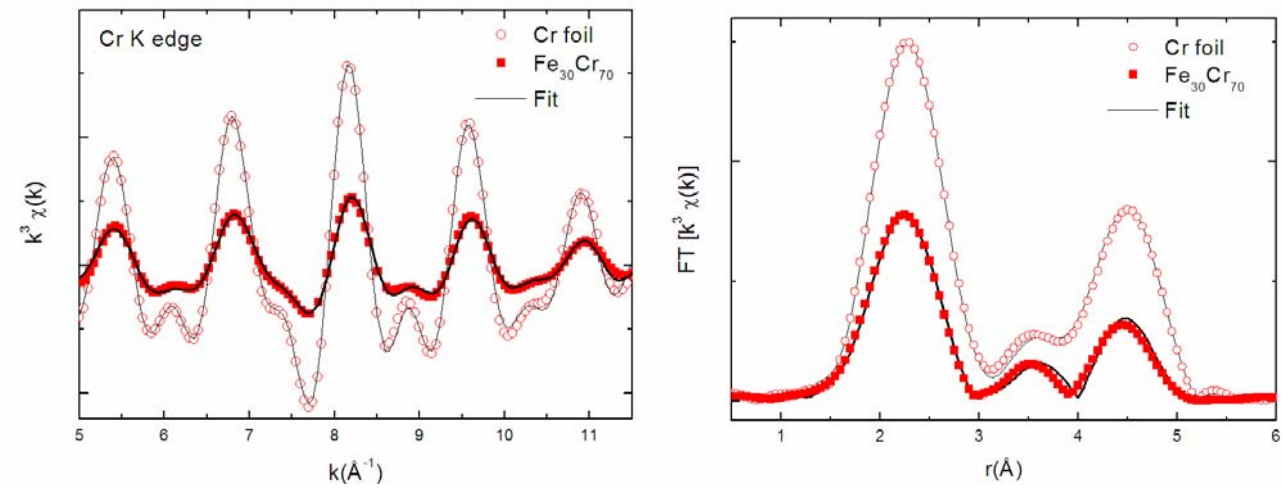

Figure 2. Left: EXAFS signal and fit of the Cr foil standard and of the $Fe_{30}Cr_{70}$ sample. Right: Fourier transform of the signals, showing the local structure in real-space.