



Herewith we present the results of the study of several iron nitrided steels samples in which it has been measured the REFLEXAFS spectra of Fe-K edge, as well as the fluorescence spectra of the alloyed elements of these steels, Cr, Mn, Mo and V K-edges, of the un-nitrided samples.

Two preliminary studies were carried out before recording the REFLEXAFS measurements, and prior to any measurements, alignment of the sample was carried out after centering the 3 circle Huber Goniometer. The reflectivity curves of the samples were measured for different incoming energies (within the range of the recorded spectrum for Fe-K edge: at 6.0 keV, 7 keV, 8 keV) to determine the value of the critical angle in each case. It was found to be very close to that of pure iron (which is 96% of the samples), although usually slightly smaller. E. g. for 8.0 keV, it is equal to 0.35 °. Moreover the roughness of the samples were checked, to minimize the loss of the intensity of the reflected wave. On the samples studied, this loss was found to be negligible.

REFLEXAFS measurements were carried out by placing the sample at an angle near (above or below) the critical angle, relative to the incident beam. A second angle ( $2\theta$ ) was used to place the detector on the maximum of the reflected beam for specular REFLEXAFS, and displaced from this angle for off-specular measurements. A set of on and off specular measurements were used to reconstruct the full layered-EXAFS measurement. This is an absolute measurement and it is necessary to measure the background (straight through beam) attenuation in order to place the reflectivity on an absolute scale. The investigated angles were 0.15, 0.20, 0.25, 0.30, 0.35, 0.40 and 0.45. During these measurements it was observed that  $2\theta$  was not twice the value of  $\theta$  (incoming beam angle), with a shift around 0.03 °. Systematic search of the possible reasons for such a shift showed that sample was not flat, showing a difference in height of around 50 micrometers between both sides. A realignment of the sample was carried out the measurements were repeated to check the affect of the lack of planarity. Additional problems to record the spectra come from the fact that the two amplifiers used for the incoming and reflected beam detectors were not identical (they were a Standford and Keithly), which complicates the data analysis due to the slightly different responses of the systems. Data are currently being analysed taking into account all these facts.

The recording of the fluorescence spectra of the alloyed elements of the un-nitrided samples has revealed very interesting information concerning the local environment of these elements: they seem to mimic the body centered structure of iron, with metal-metal distances very close to Fe-Fe distances. This is not surprising in the case of chromium, close neighbor to iron and adopting the same BCC structure, but it was unexpected for Mn, which when pure shows a completely different structure. Data analysis of these spectra are being finished and will soon be published.