ESRF	Experiment title: XAFS Study of the surface layers formed during the nitriding process of steels	Experiment number: ME-071
Beamline:	Date of experiment:	Date of report:
BM-29	from: 14 to: 22 June 2000	23/2/2001
Shifts:	Local contact(s):	Received at ESRF:
21	D.T. Bowron	
Names and affiliations of applicants (* indicates experimentalists):		
Adela Muñoz-Paez Instituto de Ciencia de Materiales de Sevilla		
	Av. Americo Vespucio s/n 41092 Seville	(Spain)
Juan Ignacio Fdez. Peruchena		
Ángel Justo Erbez		
Regla Ayala EspinarDepartamento de Química Física, Universidad de Sevilla41012Seville (Spain)		
Sofia Diaz-Moreno ESRF ID-24		

Report:

Surface treatments are one of the main tools used to control the properties of different kind of materials. One important example is the case of steels where resistance to corrosion or hardness are adapted to the required standards by modifying the outer most atomic layers by processes such as *nitriding*. In this process nitrogen atoms are introduced into the surface layers to form metal nitrides which cause a drastic increase in surface hardness. Experimentally it is intrinsically challenging to obtain information about the specific details of this process because *nitriding* only causes subtle variations in chemical composition or geometric structures. Moreover, alloyed steels are rather complex systems where minor components (<5% weight) play a fundamental role in the final mechanical properties. For this reason special techniques surface and element sensitive are required to study them.

Here we present the results of an X-ray absorption spectroscopy study carried out at station **BM29** on the inter-atomic scale effects of nitriding a commercially available steel. In the first component of this study, the changes induced by the nitriding treatments in the local structure of the chemically dilute alloyed elements, V, Cr, Mn, Mo, were monitored by measuring the fluorescence signal, I_F, of their X-ray absorption spectrum. As shown in Figure 1, it is clear that quite drastic changes in the local structure around Mo atoms were induced by the *nitriding* treatment, which causes the disruption of the metal phase with bcc structure, and

induces the formation of nitrides, which are amongst the hardest compounds after diamond. Similar changes were recorded in the Cr, V and Mn K-edge EXAFS spectra. This is the first direct structural evidence of the existence of these compounds, since alloyed elements of the steels (< 1%) and thus hardly detectable by the standard structural techniques. The sensitivity of this technique and its importance for improving our understanding of this complex system is clear when one considers a comparison with X-ray diffraction measurements. With that technique no evidence for any crystalline phases containing Mo, or any of the other alloyed elements, were found in both normal or grazing incidence investigations.



Figure 1: Fourier transform of the Mo-Kedge (E=20000 eV) EXAFS spectrum of a commercial steel (0.3% V; 3 % Cr; 0.5% Mn; 0.8% Mo; 0.3% C; rest: Fe) before (green line) and after (red line) submitting it to a gas nitriding process. The experimental data (see inset) were recorded using the ESRF 13 element Ge solid state detector to monitor the fluorescence signal with an energy resolution better than 115 eV.