

The influence of Cr-composition on the local magnetic structure of FeCr alloys

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ABSTRACT

Element specific investigations using X-ray magnetic circular dichroism (XMCD) were performed on FeCr alloys for Cr concentrations ranging from 6 to 16 at.% Cr. Measurements at the Fe *L* edge show a scattered distribution of the spin and orbital magnetic moments which is not observed for the global bulk magnetic moment. The Cr is aligned ferromagnetic up to 16 at.% Cr and in opposite direction with respect to the Fe magnetic moments.

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1. Introduction

The FeCr alloys are considered as candidate materials for structural components in advance fission reactor and future fusion power reactors, due to their superior properties of increased strength and hardness, low oxidation rate, corrosion resistance and retention of strength at high temperatures. Magnetism is inherently present in the FeCr alloy and has until recently been neglected when assessing mechanical properties and lifetime of these materials exposed to extreme conditions. *Ab initio* calculations have shown that magnetism affects the stability configuration and mobility of defects present in pure Fe [1]. Addition of antiferromagnetic Cr in the ferromagnetic Fe matrix leads to interesting magnetic configurations in the FeCr alloy phase diagram [2]. Using *ab initio* calculations, an anomaly in the formation energy of the FeCr alloy has been reported [3]. The authors found a negative value of formation energy at low Cr concentrations, which turns positive at higher Cr concentrations. Klaver et al. [4] showed that Cr–Cr repulsion promoted by magnetic frustration is significant up to the sixth nearest neighbors and very strong for the first nearest neighbors.

Recent X-ray magnetic circular dichroism (XMCD) and extended X-ray absorption fine structure (EXAFS) measurements as well as *ab initio* calculations on Fe-6.2 at.% Cr and Fe-12.7 at.% Cr have furthermore indicated the correlation existent between magnetism and structure in this transition metal alloy [5]. We intend to complement the previous work using a more complete approach

which includes further EXAFS and XMCD measurements on high purity samples, as well as density functional theory (DFT) calculations for several concentrations ranging between 5 and 16 at.% Cr. In this work we report on the XMCD measurements at room temperature for both Fe and Cr *L*-edges. Both Fe and Cr elemental moments existent in magnetically saturated specimens are investigated and compared to the measured as well as calculated global magnetic moments. We find that the Fe spin magnetic moments show a scattered distribution, which is not observed for the total bulk magnetic moment.

2. Materials and methods

FeCr materials have been prepared by arc-melting pure Fe and Cr under Ar atmosphere. The details about material preparation can be found in Ref. [5]. We have studied FeCr alloys with Cr-concentrations up to 16 at.%, as well as a high-purity Fe sample, as a reference material. The specimens were prepared as thin plates of about $4 \times 4 \text{ mm}^2$, with 0.5 mm thickness, with mechanically polished mirror like surfaces.

The XPS data were obtained with an ESCALAB 220iXL (Thermo Scientific, USA, formerly V.G. Scientific) equipment, using Al *K_α* (1486.6 eV) radiation. The specimens were cleaned by ultra-high vacuum argon sputtering operated at 3 kV, at a rate of 1 nm/min. The photoelectron spectra have been recorded at normal emission angle as a function of sputtering time, up to 900 s.

The X-ray absorption spectroscopy (XAS) and X-ray magnetic circular dichroism (MCD) experiments were performed on the Circular Polarization Beamline at Elettra Synchrotron Radiation Facility in Trieste, Italy. Absorption spectra were acquired, at room

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temperature, in total electron yield mode with an energy resolution of 0.2 eV. XMCD spectra were measured using right-handed (μ^+) and left-handed (μ^-) circularly polarized radiation, with 80% circular polarization degree and in the presence of 1.5 T magnetic field. The external magnetic field was parallel to the incident X-ray wave vector and perpendicular (out-of-plane) to the sample surface. According to bulk measurements, a field of 1 T is enough to reach the saturation magnetization. The surfaces of all specimens have been cleaned *in situ* by Ar-sputtering, using same parameters as for the XPS measurements (3 kV for more than 15 min).

The global magnetic moment was measured in all specimens using a physical property measurement system (PPMS) equipment at 10 K, with an applied field up to ± 9 T in a direction normal to the sample.

3. Results

Fig. 1 shows the Cr-2p (a) and Fe-2p (b) XPS spectra obtained on the $\text{Fe}_{0.84}\text{Cr}_{0.16}$ alloy after a sequence of Ar-sputtering cycles, ranging from 0 to 300 s. The ratio of Cr/Fe composition is shown in the lower panel for the $\text{Fe}_{0.94}\text{Cr}_{0.06}$ and $\text{Fe}_{0.84}\text{Cr}_{0.16}$ alloys, also as a function of Ar sputtering time. Computer Aided Surface Analysis for X-ray Photoelectron Spectroscopy (CasaXPS) software has been used to obtain the concentrations for Cr and Fe by appropriate background subtraction and fitting of the XPS spectra, following the procedure detailed in Ref. [6]. Sensitivity factors of 7.69 for Cr $2p_{3/2}$ and 10.82 for Fe $2p_{3/2}$ have been used to compute the elemental concentrations, which account for the electron analyzer transmission function. Impurities like C, O, Cu and Ca have been

detected before the Ar-sputtering cycles, up to 55 at.%. After 900 s of cleaning, the Ca, Cu and C peaks disappeared completely and the O content has been reduced to 5 at.%. The rest of the specimens have been Ar-sputtered using the same procedure, without a detailed analysis of compositions by XPS.

The Cr 2p (a) and Fe 2p (b) core level spectra shown in Fig. 1 have been acquired in normal emission geometry at room temperature, which corresponds to a surface sensitivity, derived from maximum electron mean-free paths, of about 2.5 nm. The specimens have not been vacuum annealed at temperatures above 900 K, where Cr surface segregation could appear. We observe that the native surfaces are dominated by the oxide signals up to 60 s sputtering time, while after 300 s, the signals from Fe^0 ($L_3 = 707$ eV) and Cr^0 ($L_3 = 574$ eV) remain. In case of Cr, only the Cr^{+3} signal is detected up to 60 s, and in case of Fe, both the Fe^{+2} ($L_3 = 710$ eV) and Fe^{+3} ($L_3 = 711$ eV) contributions give rise to the large hump seen at the high energy side of the main L_3 line.

The elemental magnetic moment contributions have been investigated using X-ray magnetic circular dichroism (XMCD) at the Fe L edge (Fig. 2) and Cr L edge (Fig. 3) for five alloys with different Cr-content ranging from 5 to 16 at.%. A high-purity Fe foil has been also measured as a reference material. The XAS and XMCD spectra plotted in Figs. 2 and 3 correspond to the $\text{Fe}_{0.84}\text{Cr}_{0.16}$ alloy only, as an illustrative example of the analysis performed for the other 5 samples but not shown here. Fig. 2 shows in the top panel the Fe $L_{2,3}$ -edge for $\text{Fe}_{0.84}\text{Cr}_{0.16}$ alloy measured in total electron yield mode (TEY), the difference of the two spectra and the integral of the difference ($\int \mu^+ - \mu^-$). The lower panel displays the sum of the two absorption spectra, the two-step function for edge subtraction and the integrated sum ($\int \mu^+ + \mu^-$) spectrum. The p , q and r

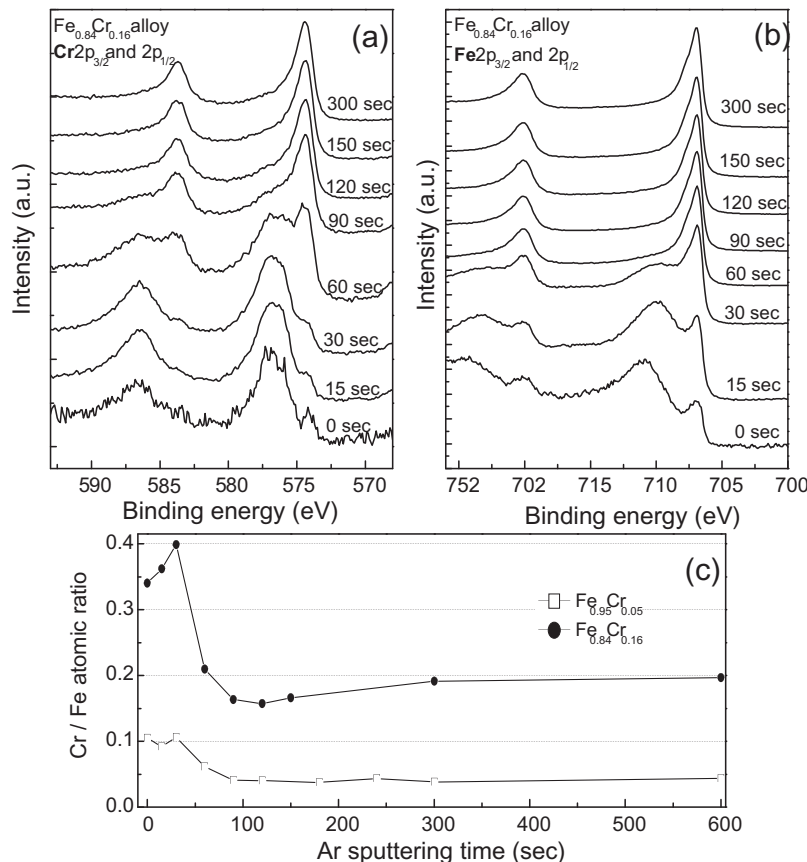


Fig. 1. (a) Cr-2p and (b) Fe-2p XPS spectra obtained on the $\text{Fe}_{0.84}\text{Cr}_{0.16}$ alloy after a sequence of Ar-sputtering cycles, ranging from 0 to 300 s. (c) The atomic Cr/Fe ratio obtained from the integrated peaks as a function of Ar-sputtering time, for the $\text{Fe}_{0.84}\text{Cr}_{0.16}$ and $\text{Fe}_{0.95}\text{Cr}_{0.05}$ alloys.

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