



Initial oxidation of polycrystalline Permalloy surface

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ABSTRACT

X-ray photoelectron spectroscopy (XPS) and work-function measurements were used in combination to investigate the initial steps of Permalloy ($\text{Ni}_{80}\text{Fe}_{20}$) oxidation at room temperature. They showed that, after oxygen saturation, the surface is covered by nickel oxide (NiO), nickel hydroxide ($\text{Ni}(\text{OH})_2$) and iron oxides (Fe_xO_y), and there is no preferential oxidation. Iron oxidation proceeds through the formation of FeO (Fe^{2+}) followed with Fe_2O_3 growth (Fe^{3+}). The oxidation is governed by a dissociative Langmuir-type oxidation: the sticking coefficient is decreasing over oxygen exposure. Oxidation continues by oxygen dissolution into the first layers to form a nano-oxide of about 8 Å in thickness.

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1. Introduction

The investigations on magnetic transition-metal thin films have seen an extensive development in recent years because of potential technical applications in magnetic devices. Characteristic features of $\text{Ni}_{80}\text{Fe}_{20}$ Permalloy (Py) thin films are soft magnetic properties, high anisotropic magnetoresistance and low magnetostriction. Therefore, Py is one of the most common materials for the magnetic data storage and can be found in a variety of magnetic micro- and nanostructures [1,2]. However, the studies about the magnetic properties of devices based on oxidized Permalloy (PyO) are scarce. Py/PyO bilayers [3] as well as PyO embedded in a Py matrix [4] are known to display exchange bias effects. This effect leads to a shift of the hysteresis loop observed in magnetometry experiment and was attributed to the existence of an AntiFerromagnetic (AFM)/Ferromagnetic (FM) coupling when the AFM thickness is sufficient to pin the FM layer. It is therefore essential to investigate the chemical species of PyO as well as the oxidation kinetics of Py.

Such investigations have already been performed, not only at low pressure and temperatures above room temperature [5,6], but also by air or during electrochemical oxidation [7,8] and plasma formation [9]. Since the final oxide strongly depends on the oxidation conditions, they have evidenced the formation of different oxides including mainly iron oxide and also nickel oxide. In their study of oxidation of a Py (100) crystal at room temperature,

Brundle et al. [10] evidenced initial iron segregation. None of these investigations have dealt with the first stages of oxidation kinetics at room temperature. The goal of the present study was to gain more insight into the controlled oxidation of polycrystalline Py thin films at low pressure and room temperature.

2. Experimental details

2.1. Experimental set-up

The Py thin films were grown on Si (100) substrate by radio-frequency (RF) diode sputtering performed with the standard Z550 Leybold equipment. A DC magnetic field of 300 Oe was applied along the film plane during deposition in order to induce a well-defined uniaxial anisotropy. The background pressure was better than 3×10^{-7} mbar. The (111) texture of fcc thin films was checked by X-ray diffractometry with $\text{CuK}\alpha$ radiation. The samples were transferred to ultrahigh vacuum (UHV) electron analysis chamber (base pressure of about 5×10^{-10} Torr). The native oxide was removed by using a 1 keV Ar^+ ion beam. We checked by XPS that the Ni/Fe ratio showed no preferential sputtering effect.

Oxidation was performed at room temperature by exposure of the sample to an O_2 partial pressure of 10^{-7} Torr from 0 to 5000 L ($1 \text{ L} = 10^{-6} \text{ Torr s}$). It was investigated through XPS and UPS measurements. The XPS chamber contains an X-ray source positioned at 5° with respect to the surface normal and a cylindrical mirror analyser (CMA) from Riber in order to allow angle-integrated measurements with an analysis resolution of about 1 eV.

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The take-off angle of the collected electrons with respect to the surface is about 45°. Mg K α radiation was used to avoid coincidence between the Ni Auger peaks caused by the Al K α irradiation and the Fe 2p emission. All spectra were calibrated using the C 1s peak with a fixed value of 284.8 eV. The surface work-functions were measured from the high binding-energy cut-off of Ultraviolet Photoelectron Spectroscopy (UPS) spectra. Biasing of the sample (8 V negative on the sample) enabled us to decrease the secondary electron peak at high-binding-energies with no effect on spectral features. UPS experiments were carried out with Ultraviolet photons (HeI 21.2 eV), produced by a helium cold-cathode gas discharge. The angle of incidence of the probe beam was 45° with respect to the surface normal, and the electrons were collected at right angle to the surface. Moreover, the experiments on pure nickel and pure iron thin films were performed under the same experimental conditions to allow comparison with Py.

2.2. XPS fitting procedure

To gain more information into the chemical states of nickel, iron and oxygen ions, our spectra of the Fe 2p, Ni 2p and O 1s core levels were curve-fitted with a non-linear least square fitting program (XPSPeak 4.1 version [11]).

The first important step for quantitative analysis of XPS spectra is the correction of secondary electron background. The most current background correction is the well-established and easy to use Shirley method [12]. Sherwood and co-workers [13] concluded that the Shirley background subtraction is the most effective method in fitting the short energy range found in typical core XPS fits. A second approach is the formalism of Tougaard [14], which relies on rigorous modeling of extrinsic losses due to inelastic scattering of electrons associated with their transport through the solid. Recently, by comparison of intrinsic zero-energy loss and Shirley-type background corrected spectra of iron oxides, Oku et al. [15] demonstrated the validity of Shirley background use. In the present investigations, Shirley background was subtracted from each spectrum before the peak fitting.

The curve fitting was aimed at resolving the overlap between peaks corresponding to different chemical states, and so at finding the area and binding-energy relative to each component of the spectrum. The metal and oxide states were both represented with a mixed Gaussian-Lorentzian sum function. A characteristic feature of metallic states is an asymmetric lineshape resulting from the continuum of possible final states caused by interaction between the core hole and the valence electrons [16]. In the case of oxide states, the lineshape is also asymmetric and broadened because of the multiplet splitting caused by the interaction between the 2p electrons and the unpaired 3d electrons and at the origin of a multitude of possible final states [17]. Consequently, the metal and oxide lineshapes of the 2p spectra were taken into consideration by using an asymmetric peak function, except for the nickel oxide 2p_{3/2} envelop, which was fitted by using two symmetric peaks [18].

The binding-energy, full width at half maximum (FWHM), Gaussian-Lorentzian ratio (GL), and asymmetry parameters (TS for tail scale factor and TL for tail length at half width at half maximum of the peak) for the spectra components were determined by using spectra recorded from clean iron and nickel samples and powder samples of FeO, Fe₂O₃ and Fe₃O₄. For oxidized nickel, we used the results reported by Laksono et al. [18] to examine the nickel oxidation. For the 2p region, the intensity of each 2p_{1/2} component was fixed at half the intensity of the 2p_{3/2} one, and the asymmetry parameters and the GL ratio of the 2p_{3/2} and 2p_{1/2} main peaks were set equal. Then, the curve fitting parameter values (see Table 1) were all kept constant and used in the analysis of oxidized Permalloy sample.

Table 1

Curve fitting parameters for O(1s), Ni(2p_{3/2}) and Fe(2p_{3/2}) XPS spectra

	BE (eV) ^a	FWHM (eV)	GL (%)	TS/TL	I _{sat} /I
O 1s					
O ²⁻ (NiO)	529.5	1.7	100	Symmetric	
O ²⁻ (Fe _x O _y)	530.0	1.7	100	Symmetric	
OH ⁻	531.2	1.7	100	Symmetric	
Ni 2p_{3/2}					
Ni ⁰	852.8	1.8	50	0.1/70	
Ni ⁰ sat	858.8	3.6	50	Symmetric	0.20
Ni _{ox} ²⁺	854.1	2.5	70	Symmetric	
Ni _{ox} ²⁺ doublet	855.9	3.5	70	Symmetric	0.99
Ni _{ox} ²⁺ sat	861.2	4.0	70	Symmetric	0.70
Ni _{hyd} ²⁺	855.8	3.3	70	0.1/20	
Ni _{hyd} ²⁺ sat	861.6	3.4	70	Symmetric	0.30
Fe 2p_{3/2}					
Fe ⁰	707.3	2.3	50	0.3/200	
Fe _{ox} ²⁺	709.4	2.9	70	0.2/150	
Fe _{ox} ²⁺ sat	715.0	3.7	70	Symmetric	0.05
Fe _{ox} ³⁺	710.7	3.1	70	0.2/150	
Fe _{ox} ³⁺ sat	719.1	4.2	70	Symmetric	0.05
Fe _{hyd} ³⁺	712.2	3.9	70	0.2/150	

^a Uncertainty \pm 0.1 eV.

3. Results and discussion

3.1. XPS results

Fig. 1 displays the O(1s) binding-energy spectra for different oxygen exposures at room temperature. An asymmetric structure grows with oxygen exposure at about 530 eV. This signal mainly corresponds to oxygen atoms in the nickel oxide NiO (O²⁻) at oxygen saturation (3100 L). This value is in agreement with the data reported in the literature for the formation of NiO (529.60 \pm 0.15 eV [19,20]). Moreover, an enhancement can be seen in the high binding-energy part of the structure: it is well explained by the presence of hydroxide (OH⁻) ions (531.2 eV), whose adsorption is known to stabilise NiO oxide [20]. This species is issued from the dissociation of residual water in the vacuum chamber. In agreement with previous data on nickel [20,21], the UPS and XPS (532.4 eV [22]) spectra show neither molecular water, nor molecular oxygen. Then, the oxidation proceeds by dissociative

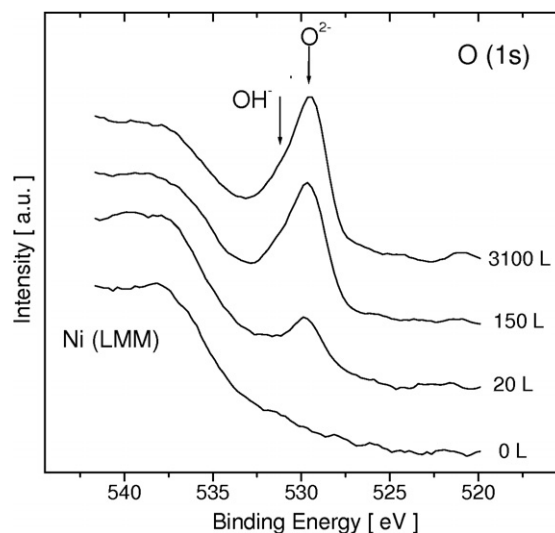


Fig. 1. XPS spectra of the O(1s) region from a Permalloy surface exposed to the indicated amount of oxygen. Arrows indicate the positions of nickel oxide and hydroxide species.

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