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Experimental determination of gadolinium scattering characteristics in neutron reflectometry with reference layer

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ABSTRACT

The experimental phase determination in neutron reflectometry with gadolinium reference layer demands precise values of the parameters of reference layer including its scattering characteristics. It is especially important for time-of-flight experiments since in this case one must know these characteristics at each reflectivity point. So it is desirable to specify the reference layer scattering parameters directly during the experiment. In this work the way for determination of scattering characteristics, using the neutron absorption measurements on a thin film sample, is presented.

1. Introduction

Neutron reflectometry is a powerful method of studying the planar heterostructures and their interfaces by analyzing the specular reflection of low-energy neutrons beam, which is incident at small glancing angles to the sample surface. Neutron reflectometry can be applied to the study of magnetic media; in this case, polarized neutrons or combinations of different methods are used. The modern review of advances of polarized neutron reflectometry for studying magnetic nanostructures using the combination of small angle specular reflection together with off-specular scattering is given in Ref. [1]. Applying the neutron off-specular scattering for the investigation of magnetic ordering in exchange-coupled multilayers was demonstrated in Refs. [2,3]. The polarized neutron reflectometry method is used to investigate the distribution of nuclear neutron-optical potential along the normal to the sample surface at depths of up to several thousand angstroms. The objects of study are the surfaces of bulk solids, thin films on substrates, the surfaces of the liquids and the interfaces in heterogeneous systems. Polarization reflectometry allows us to analyze the depth dependence of the local magnetization vector.

The model fitting procedures are difficult to use for analyzing samples with complicated chemical and magnetic structures due to big number of model parameters. In this case the optimization algorithms works worse and often obtain the solution without physical meaning. It is more preferable to use model-independent methods which allow analyzing experimental data without a priori parameters choice and their subsequent refinement. As a rule, such methods use the solution of Gelfand-Levitan-Marchenko equation [4] and it is necessary to know the phase of a complex reflection coefficient which cannot be obtained in experiment.

In our previous work we have shown the possibility of using the gadolinium as a reference layer in neutron reflectometry [5] and presented an approach for experimental determination of complex reflection coefficient [6]. Commonly, the neutron reflectometers work in time-of-flight mode so it is necessary to know the gadolinium scattering characteristics over the full available range of wavelengths. One can use the theoretical values [7], but it is more preferable to determine these parameters in experimental way for the sample of gadolinium, which was used for a reference layer synthesis. Thus one can to take into account possible variations of isotope composition, which can affect the resonance scattering characteristics. Polarized neutron diffraction measurements from the gadolinium film have already been attempted [8]. In that work the real and imaginary parts of the magnetic scattering length were calculated and several points for the real part were experimentally obtained.

In this paper we present results of determination the complex scattering length density for thin gadolinium film in a wide range of neutron wavelengths. The scattering characteristics calculated from neutron absorption data. Such experiment can be performed directly before reflectivity measurements for samples with reference layers.

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2. Methodology

In quantum mechanics the coherent scattering length b is described as sum of Lorentz functions for all possible resonances (relativistic Breit-Wigner distribution) [9]:

$$b = R' + \sum_j c_j \cdot \frac{\tilde{\lambda}_j \Gamma_{nj}}{2(E - E_j) - i\Gamma_j} \quad (1)$$

Here c_j is the resonance weight related with the content of respective isotope in the gadolinium sample, E – neutron energy, E_j – resonance energy, Γ_{nj} – neutron width of resonance peak, Γ_j – total width of the resonance peak (sum of neutron width and radiative width). The reduced wavelength $\tilde{\lambda}_j$ corresponding to the resonance is:

$$\tilde{\lambda} = \frac{\hbar}{\sqrt{2mE_j}}. \quad (2)$$

The non-resonance contribution into scattering length for Gd isotopes can be expressed in terms of mass number A [7]:

$$R' = 1.16A^{1/3} + 0.6(\text{fm}) \quad (3)$$

But this is not a general relation for all non-resonant coherent scattering lengths as it represents only the radius contribution to the neutron-nuclear interaction.

The real and imaginary parts of (1):

$$b_r = R' + \sum_j c_j \cdot \frac{2\tilde{\lambda}_j \Gamma_{nj}(E - E_j)}{4(E - E_j)^2 + \Gamma_j^2}$$
$$b_i = \sum_j c_j \cdot \frac{\tilde{\lambda}_j \Gamma_{nj}\Gamma_j}{4(E - E_j)^2 + \Gamma_j^2} \quad (4)$$

Since the two gadolinium isotopes (Gd^{155} и Gd^{157}) have resonance absorption, the sums in (4) will consist of two components.

On the other hand, the imaginary part b_i can be related to the absorption cross σ [9]:

$$b_i = \frac{\sigma}{2 \cdot \lambda} \quad (5)$$

whereas σ can be calculated from experimental data:

$$\sigma = -\frac{\ln\left(\frac{I - I_b}{I_0 - I_b}\right) \cdot \cos\alpha}{n \cdot d}. \quad (6)$$

Here I – intensity of transmitted beam, I_b – intensity of background radiation, I_0 – intensity of incident direct beam, α – angle between sample surface normal and incident beam, n – gadolinium atomic density, d – thickness of the sample.

Thus, one can perform the experiment on neutron absorption through gadolinium film, obtaining I, I_b and I_0 in a certain range of wavelengths and calculate experimental dependence $b_i(\lambda)$ using (6) and (5). Then it is possible to refine gadolinium scattering parameters in (4) with any method of optimization. The real part can be calculated using (4) with obtained parameters or by Kramers-Kronig relations.

3. Experiment

The sample represents a thin gadolinium film (thickness is 5000 Å) on silicon substrate (30 × 30 mm). The structure formula is Si/Cr (100 Å)/Gd(5000 Å)/Cr(100 Å). The first Cr layer is a buffer; the upper layer is a cap preventing gadolinium oxidation. The sample has been prepared by magnetron sputtering unit «ULVAC» in the Institute of metal physics (Ekaterinburg, Russia).

The intense neutron absorption by gadolinium is due to presence of two isotopes - Gd^{155} and Gd^{157}. These nuclei have a resonance interaction with thermal neutrons [4]. The silicon and chromium absorb neutrons much more weakly, approximately in 1000 times. Thus their influence is negligible and one can consider the sample as gadolinium film only.

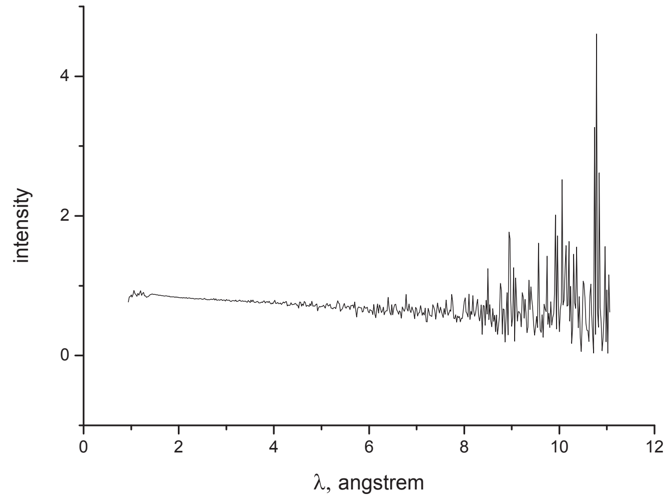

Fig. 1. Intensity of neutron transmission through a film Si//Cr(100 Å)/Gd (5000 Å)/Cr(100 Å).

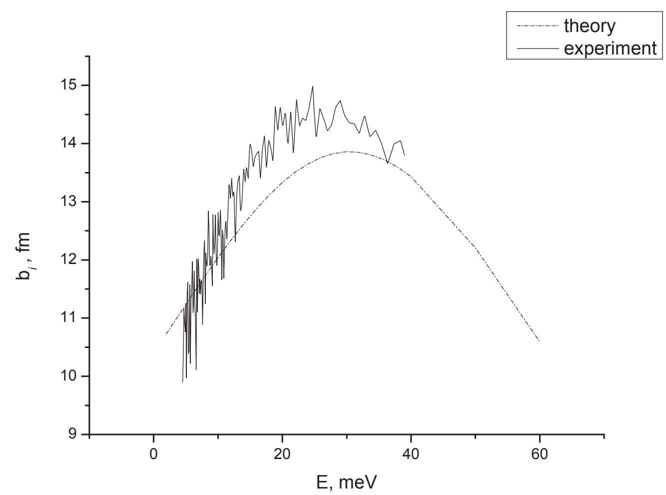

Fig. 2. Experimental and theoretical scattering lengths (imaginary parts).

Table 1
Experimental parameters of resonant neutron absorption by gadolinium isotopes.

Parameter	Experimental value	Theoretical value [8]
Resonance energy (Gd^{155}), eV	0,0192	0,0268
Neutron width (Gd^{155}), eV	6,84E-05	6,52E-05
Radiative width (Gd^{155}), eV	0,107	0108
Isotope content (Gd^{155}), %	14,7	14,8
Resonance energy (Gd^{157}), eV	0,0323	0,0314
Neutron width (Gd^{157}), eV	3,06E-04	2,94E-04
Radiative width (Gd^{157}), eV	0,102	0106
Isotope content (Gd^{157}), %	15,14	15,65

The neutron absorption spectrum was obtained at instrument REFLEX-P of IBR-2 pulsed reactor (Joint Institute for Nuclear Research, Russia, Moscow region, Dubna). REFLEX-P is the time-of-flight neutron reflectometer situated on the 9-th beamline. The available neutron wavelength band is from 1 to 12 Å. Scattered neutrons registration was carried out by 2D He^3 detector with 200 × 200 mm^2 sensitive area with 1.5 mm spatial resolution. The reflectometer provides measurement in Q range from 0.001 to 0.25 $Å^{-1}$. The exposure time was 125 min for all measurements – direct beam, transmitted beam and background radiation. Data processing was performed in a smaller wavelength range from 0.9 to 6 Å, since the spectrum has considerable statistical noise