Effect of the State of Interlayer Boundaries on the Magnetoresistive Properties of Layered Co/Cu Nanostructures

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Abstract
The influence of the state of interfaces on the magnetoresistive properties of Co/Cu superlattices has been studied by the methods of nuclear magnetic resonance and X-ray reflectometry. It has been found that the magnetron-sputtered superlattices with the highest value of the giant magnetoresistance effect have the largest fraction of highly perfect Co/Cu interlayer boundaries, as well as practically the smallest fraction of cobalt atoms localized in the interfaces.

1 Introduction

Interest in exchange-coupled superlattices with the effect of giant magnetoresistance remains due to their practical application in modern magnetically sensitive devices of radio electronics and spintronics [1, 2]. Superlattices are alternating layers of magnetic 3-d metals (or alloys based on them) and non-magnetic metals. It is known that Co/Cu superlattices have a sufficiently high value of the giant magnetoresistance (GMR) effect [3–5]. For example, the magnitude of the magnetoresistive effect reaches ~65% at the first antiferromagnetic maximum at room temperature [3]. Structural diagnostics of superlattices is an urgent problem, since the magnetic and magneto-transport properties of magnetic metallic superlattices depend on the structural features of the layers and interlayer boundaries (interfaces).

Mössbauer spectroscopy provides great opportunities for determining the state of layers and interfaces [6], but the application of this method is limited because the Fe layer (preferably enriched in the $^{57}$Fe isotope) is required for the research. Another well-proven method for studying the state of interfaces is the X-ray reflectivity
The problem is that a significant number of parameters are varied when modeling reflectograms, which complicates the interpretation of experimental data. Accordingly, to ensure obtaining a reliable result in the assessment of the state of interlayer boundaries, it is advisable to use an alternative method. Earlier, the method of nuclear magnetic resonance was successfully applied to study the structural features of layers and interfaces in metallic magnetic Co/Cu-based superlattices [8–12].

The main idea of application the NMR method in the study of multilayer Co/Cu structures is as follows. Due to hyperfine interaction, magnetic moments of cobalt atoms create local magnetic fields at the location of $^{59}$Co nuclei, the magnitude and direction of which significantly depend on the magnetic and structural features of the nearest environment of the probe nucleus. The NMR method allows investigation of these local fields distribution in a sample, and therefore is a powerful method for studying the structural characteristics of multilayer nanostructures.

In accordance with [13–16], increasing the number of pairs of layers leads to increasing of the effect of giant magnetoresistance. However, our earlier studies [17] have shown that an increasing the number of bilayers leads to decreasing of the GMR effect. This was associated with a decrease in the proportion of highly perfect boundaries, since interfaces are the centers of scattering of conduction electrons [18]. Possible reason might be related with the non-optimal sputtering mode.

In this work, the problem was posed to estimate the fraction of highly perfect interlayer boundaries, the roughness of the interfaces, and their effect on the magnetoresistive properties of exchange-coupled Co/Cu superlattices. These superlattices are the result of the development of magnetron sputtering technology and, accordingly, have high functional characteristics and magnetoresistance.

2 Experimental Materials and Methods

Multilayer Co/Cu structures were fabricated in Ulvac MPS-4000-C6 sputtering system by DC magnetron sputtering on glass substrates (Corning). Before deposition the substrate surface was cleaned by ion etching in an argon atmosphere in a deposition system. The material of the protective layer is Cr 5 nm thick. The thickness of the PyCr buffer layer (PyCr = (Ni$_{80}$Fe$_{20}$)$_{40}$Cr$_{60}$) is 5 nm. This buffer layer is used to form a more perfect crystal structure of the layers [18, 19].

Superlattices were deposited at room temperature of the substrate, at fixed argon pressure of 0.1 Pa and magnetron power of 100 W. These conditions made it possible to sputter target materials Co, Cu, Cr, PyCr at rates of 2.7, 3.0, 6.9, 3.0 and 3.3 nm/min, respectively. The structural formula of the samples is as follows: glass//PyCr(5 nm)//[Co(1.5 nm)/Cu(1 nm)]$_n$/Cr(5 nm), $n = 10, 12, 14, 16$ and 20.

X-ray studies of the samples were carried out using the Philips Empyrean hardware-software complex. The reflection and diffraction patterns were recorded in Co$K_{\alpha}$ radiation. Modeling of reflectograms (XRR) was performed using the Pana lytical X’Pert Reflectivity software. The technique and features of modeling of XRR reflectograms are described in [17].
The spectra of the samples under study were obtained by the method of nuclear magnetic resonance on $^{59}\text{Co}$ nuclei in the frequency range 140–240 MHz. The NMR spectra were recorded on a pulsed NMR spectrometer at liquid helium temperature (4.2 K) in a local magnetic field without external magnetic field. The spin echo signal is formed by a sequence of two coherent radio-frequency pulses $(\tau_p)_x - t_{\text{del}} - (\tau_p)_y - t_{\text{del}} - \text{echo}$, which create an alternating magnetic field with an amplitude of the circular component $H_1 \approx 10$ Oe in the resonant coil with the sample. The duration of the pulses $\tau_p$ was 0.5 $\mu$s, and the time interval $t_{\text{del}}$ between pulses was 9 $\mu$s. The frequency step was 1 MHz.

According to the previously described method and the model used, the NMR spectrum was decomposed into several Gaussians, each of which corresponds to a $^{59}\text{Co}$ probe nucleus with a certain type of nearest environment [12].

The magnetoresistance was measured according to the standard technique by the four-contact method at DC with current flowing in the plane of the layers. All measurements were taken at room temperature. The magnetoresistance was determined by the formula: $MR = (R(H) - R_s)/R_s$, where $R_s$ is the resistance in the magnetic saturation field.

## 3 Results and Discussion

The magnetoresistance dependence on the number of pairs of layers, $n$, of superlattices is shown in Fig. 1. Figure 1 demonstrates the increasing of magnetoresistance with increase in the number of bilayers for Co/Cu superlattices prepared by the method of magnetron sputtering that is consistent with [13–16].

The studies have shown that in the Co/Cu superlattices under study the maximum magnetoresistance is $\Delta R/R_s = 60.2\%$, and the magnetic saturation field is $H_s = 15$ kOe for 16 pairs of Co/Cu layers (Fig. 2).

![Magnetoresistance dependence on the number of bilayers in superlattices glass/PYCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]$_n$/Cr(5 nm)](image)
Figure 3 demonstrates the X-ray diffraction pattern of superlattices glass//PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]\textsubscript{n}/Cr(5 nm) with \(n = 10, 12, 14, 16\) and 20. The diffraction pattern contains only one line, which is responsible for reflection from \{111\} planes. Thus, an axial texture with the \(<111>\) axis perpendicular to the plane of the substrate is formed in the sample under study.

Figure 4 shows the results of low-angle X-ray diffraction of superlattices glass//PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]\textsubscript{n}/Cr(5 nm) with \(n = 10, 12, 16\) and 20. It can be seen that the first Bragg peak for all nanostructures is located near the angle \(2\theta = 4.35^\circ\), which means that the superstructure periods are equal. The presence
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The roughness of the layers was determined as the geometric roughness, the numerical value of which is obtained by calculating the root-mean-square roughness [20].

Modeling of reflectograms has shown that the interface roughness does not directly depend on the number of bilayers and changes in the region from $\sigma = 0.17$ nm for $n = 12$ to $\sigma = 0.42$ nm for $n = 20$. (Fig. 5).

**Fig. 4** X-ray reflection patterns of superlattices glass//PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)],/Cr(5 nm), $n = 10, 12, 16, 20$. Co $K_{\alpha}$ radiation

**Fig. 5** Dependence of roughness of Co/Cu interfaces on the number of bilayers in superlattices glass//PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)],/Cr(5 nm), for $n = 10, 12, 16, 20$
Thus, it can be concluded that there is no correlation between the number of bilayers and the roughness of the interfaces. Note that for a superlattice with \( n = 16 \) having the highest magnetoresistance MR = 60.2%, the roughness of the interlayer boundaries is \( \sigma = 0.28 \) nm.

To understand the term “highly perfect” interface, let us simulate the crystal lattice of a periodic structure in the region of the interlayer boundary. Figure 6 shows the result of modeling the Co/Cu interface of a superlattice with an axial structure \(<111>\) perpendicular to the plane of the layers, in the projections from the side (Fig. 6a) and from above (Fig. 6b). In Fig. 6a, the upper atomic layer is Cu (orange balls), and the lower atomic layer is Co (blue balls). For the central atom of the cobalt layer, the bonds with atoms of the nearest environment in the copper layer are shown. It can be seen from the figure under discussion that there are only three nearest neighbors for the \(^{59}\)Co probe nucleus in the atomic layer of copper. It is this configuration that makes it possible to obtain a highly perfect Co/Cu interlayer interface and, as a consequence, the minimum value of the probability of electron scattering at the interface, which leads to the maximization of the GMR effect. The same element of the crystal lattice, but in the projection from above, is shown in Fig. 6b.

Figure 7 shows the normalized NMR spectra of the entire series of samples. It can be seen from the figure that the spectra are principally similar: the same spectral line width at 216 MHz (there are no copper atoms in the first coordination sphere of the probe nucleus), and the position of this line also does not change depending on the number of bilayers. The rest of the spectral lines (corresponding to a different number of copper atoms in the immediate environment of the probe nucleus) visually coincide in width and resonance frequency.

Figure 8 demonstrates an example of the NMR spectrum modeling of the superlattice glass//PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]\(_{20}\)/Cr(5 nm). The following parameters were varied when modeling the spectrum: the line width (the same for all lines), the position of the peaks, as well as their intensities. In Fig. 8, the individual spectral lines are indicated by dashed lines. The resulting spectrum is shown as a solid line. The deviation of the resonance frequency of each spectral line does not exceed \( \sim 1.5 \) MHz, which is a fairly good result of agreement

![Co/Cu interface model for superlattice glass//PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]\(_{20}\)/Cr(5 nm) with axial texture \(<111>\) perpendicular to layers plane: side projection (a) and above projection (b)](image)
between the theoretical model and experimental data. The frequency of the most intense resonance line is 216 MHz, which is close to the value obtained for bulk Co (217 MHz) [17]. Consequently, this line is formed by Co atoms located in the bulk of the layers, which have the FCC structure. The resonance line at 228 MHz, which appears in the presence of the HCP modification of Co, is absent, indicating that there is no HCP modification of Co and stacking faults in the samples under study.

![Fig. 7 NMR spectra of metal nanostructures in the range of 140–240 MHz for glass/PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]\(_n\)/Cr(5 nm), \(n = 10, 12, 14, 16, 20\). Insert. NMR spectra of superlattices glass/PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]\(_{20}\)/Cr(5 nm) (white circle) and Al\(_2\)O\(_3\)/Nb(3 nm)Cu(2 nm)/[Co(1.5 nm)/Cr(0.9 nm)]\(_{20}\) (black star) [21]](image)

![Fig. 8 Modelling of NMR spectrum of superlattice glass/PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]\(_{20}\)/Cr(5 nm)](image)
Designations of Gaussian $i$ correspond to the different types of nearest environment of the probe nucleus, where index «$i$» is the number of copper atoms in the coordination of the probe nucleus. For example, $I_0$ represents that zero copper atoms take place in the coordination of cobalt, thus the probe nucleus is localized in cobalt layer volume. Line $I_3$ corresponds to cobalt atoms with three copper atoms in nearest environment. Substitution of one cobalt atom for one copper atom in the nearest environment leads to a decrease in the value of the hyperfine field (HFF) at the probe nucleus and, consequently, to a decrease in the resonance frequency by 16–18 MHz [22, 23]. Thus, resonance lines with a frequency lesser than 216 MHz correspond to cobalt atoms localized in the interface region.

To compare the state of interlayer boundaries, the NMR spectrum of a Co/Cu superlattice with the structural formula $\text{Al}_2\text{O}_3//\text{Nb}(3 \text{ nm})\text{Cu}(2 \text{ nm})/\{\text{Co}(1.5 \text{ nm})/\text{Cr}(0.9 \text{ nm})\}_20$ prepared by the method of molecular beam epitaxy (MBE) was taken from [21] and simulated according to the technique used to simulate the NMR spectra of the investigated series of superlattices. Both multilayer superstructures have the <111> texture. The inset in Fig. 7 shows the NMR spectra (normalized to unity) for superlattices glass//PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]$_{20}$/Cr(5 nm) and $\text{Al}_2\text{O}_3//\text{Nb}(3 \text{ nm})\text{Cu}(2 \text{ nm})/\{\text{Co}(1.5 \text{ nm})/\text{Cr}(0.9 \text{ nm})\}_20$ [21]. It can be noted that the width of the resonance line at 216 MHz is practically identical for both spectra. This suggests that superlattices prepared by magnetron sputtering have a high degree of structural homogeneity within the Co layer. The NMR spectrum of the superlattice prepared by the MBE method contains two resonance lines: ~216 MHz (a probe core with no copper atoms in its immediate environment—inside the Co layer) and ~168 MHz (a probe core with three atoms of Cu).

The internal structure of interlayer boundaries can be characterized by the proportion of areas of "perfect" conjugation in the total interface surface. The presence of the <111> texture means that the "perfect" conjugation corresponds to the situation when the boundary coincides with the {111} crystallographic plane. In this case, each Co atom located at the boundary corresponds to three Cu atoms in the nearest environment.

The resonance line at a frequency of ~168 MHz is formed by the nuclei of Co atoms of a highly perfect Co/Cu interface, which coincides with the close-packed <111> plane of the FCC lattice, when each Co atom has three Cu atoms in the nearest environment. Consequently, the fraction of interface atoms surrounded by three copper atoms, and hence the fraction of highly perfect interlayer boundaries, can be determined as $I_3/\sum_i I_i$, where $i$ corresponds to the number of copper atoms in the immediate environment of the probe nucleus. Modeling of NMR spectra makes it possible to quantify the proportion of highly perfect boundaries (Fig. 9).

For zero-field NMR in magnetically ordered systems lines' integral intensities are not proportional to number of the atoms due to enhancement factors (EF) which may differ for different Co positions. To understand differences between enhancement factors for cobalt NMR lines $I_0$, $I_1$, $I_2$, $I_3$, $I_4$ the dependence of amplitude of NMR signal on the radiofrequency field amplitude [24, 25] was measured. It was found that optimal power of the radiofrequency field for NMR lines $I_0$, $I_1$, $I_2$, $I_3$, $I_4$ differed by no more than 10%. Thus, we concluded, that
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The enhancement factor for all NMR lines are close. Consequently, using of lines’ integral intensities for quantitative estimates is correct.

According to Fig. 9, the proportion of highly perfect interlayer boundaries varies in the range from 38 to 46%. For a superlattice with \( n = 16 \), which has the greatest GMR effect, the fraction of highly perfect boundaries is 46%. In the superlattice from [21], only highly perfect interlayer boundaries are formed, which is probably due to preparation by molecular beam epitaxy.

The state of interlayer boundaries can also be characterized by the fraction of Co atoms localized at the interfaces. The fraction of such atoms can be determined by the ratio of the total intensity of resonance lines formed by atoms located in the interfaces to the total intensity of all resonance lines, that is \( \frac{\sum_{i=1}^{4} I_i}{\sum_{i=0}^{4} I_i} \), see Fig. 10, columns. Figure 10 also demonstrates the fraction of Co atoms involved in the formation of highly perfect interfaces: \( \frac{I_3}{\sum_{i=4}^{4} I_i} \) for superlattices glass//PyCr(5 nm)/[(Co(1.5 nm)/Cu(1 nm))]_{20}/Cr(5 nm) (shaded part of columns) and Al_{2}O_{3}//Nb(3 nm)Cu(2 nm)/[(Co(1.5 nm)/Cr(0.9 nm))]_{20} [21] (solid horizontal line).

Modeling of NMR spectra made it possible to establish that the fraction of cobalt atoms forming interlayer boundaries varies from 28 to 38%. The absence of a significant change in the number of cobalt atoms localized in the interface region is in agreement with the data on similar magnetic superlattices CoFe/Cu [20]: an increase in the coherent scattering region in the direction perpendicular to the film plane with an increase in the number of bilayers \( n \). Note that in the case of a superlattice with MR = 60.2%, the fraction of Co atoms localized at the interfaces is 29%. Figure 10 shows that the number of atoms forming highly perfect boundaries varies from 10 to 13% and practically coincides with the number of atoms forming highly perfect boundaries in a superlattice fabricated by the MBE method. Consequently, the fraction of atoms that do not form highly perfect interfaces characterizes the degree of roughness of the interlayer boundaries. Since this quantity is not zero, we can...
conclude that interlayer boundaries of the diffuse type are formed in the superlattices under study.

4 Conclusion

Structural features of glass/PyCr(5 nm)/[Co(1.5 nm)/Cu(1 nm)]_n/Cr(5 nm) superlattices with n = 10, 12, 14, 16 and 20, and an effect of the state of Co/Cu interfaces on their magnetoresistive characteristics have been investigated by nuclear magnetic resonance, X-ray diffraction, and X-ray reflectometry.

It has been found that in the superlattices under study, fabricated by the proven laboratory technology of magnetron deposition, the fraction of highly perfect interfaces varies from 38 to 46%, and the fraction of cobalt atoms forming interlayer boundaries varies from 28 to 38%. It is shown that diffuse interlayer boundaries are formed in the nanostructures under study. It has been revealed that the fraction of cobalt atoms localized in highly perfect interfaces for the investigated superlattices prepared by the magnetron sputtering and for the superlattice from [21] practically coincides, which is an interesting result that deserves a more detailed study.

Increasing the number of bilayers leads to worsening of interfaces and decreasing GMR effect [17]. In present paper, we have shown the possibility to keep high quality of the interfaces. It is possible to increase GMR effect by increasing the number of bilayers in accordance with [13–16].

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Declarations

Competing interests The authors declare no competing interests.

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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