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Transport properties of Al/Ni and Al/Ag multilayer systems

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Abstract. The electrical resistivity, Hall effect, magnetoresistance and thermoelectric power of Al/Ni and Al/Ag sputtered multilayer films have been measured between 1.6 K and 300 K. The temperature dependence of the electrical resistivity of Al/Ni samples with small layer periodicity shows a negative temperature coefficient at low temperatures and saturation above about 100 K. The Hall coefficient of both systems depends sensitively on their layer periodicity. A reduction of ferromagnetic moment in small wavelength Al/Ni samples was clearly observed in the Hall effect and the magnetoresistance. The possibility of some formation of intermetallic compounds in these materials is discussed based on the effect of annealing on the transport properties. The measurement of the Hall effect and the magnetoresistance in multilayer systems gives various information about their magnetic properties.

1. Introduction

The measurement of transport properties of intermetallic systems has already been a fruitful field of investigation. Many experimental studies have been made on the magnetic and superconducting properties of metallic multilayer systems [1, 2]. Since the discovery of the giant magnetoresistance in Fe/Cr multilayer systems [3–5], an increasing number of magnetoresistance measurements are being carried out on metallic multilayer systems which include at least one ferromagnetic component.

A few measurements of the Hall effect in multilayer systems have been reported [6–8]. Since the anomalous Hall effect is very sensitive to the magnetic state of surface regions [9], it is also expected to be effective for the investigation of magnetic multilayer systems. For instance in Ag/M (M = Ni, Co or Fe) multilayer systems, the effect of surface anisotropy on the anomalous Hall effect has been clearly demonstrated [6].

The thermoelectric power has been investigated in a very limited number of systems and very few effects characteristic of multi-layering have been reported.

In this work, the precise measurements of the electrical resistivity, the thermoelectric power, the Hall effect and the magnetoresistance below 300 K have been made on a

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series of samples with different layer periodicities (Λs). We chose two multilayer systems Al/Ni and Al/Ag for which the formation of intermetallic phases between the components is possible. We study several points of interest: (i) the effect of lattice mismatch—the Al/Ni system has large lattice mismatch, while Al and Ag have the same crystal structure with almost equal lattice constants; (ii) the effect of mutual solubility—we can compare Al/Ni with Ag/Ni [6] in which the two components are completely insoluble in each other; (iii) the influence of the non-magnetic component on the Ni magnetic moment—in contrast to Mo/Ni or Cu/Ni systems, little reduction in the magnetic moment of Ni in the Ag/Ni system has been observed as Λ decreases.

2. Experiment

The samples were made by DC triode sputtering with magnetic confinement at room temperature without controlling the substrate temperature [10]. In this work, the thicknesses of both components are always equal for all Λ. The base pressure was better than 10^{-8} torr and the pressure of argon in the sputtering process was 3 to 15 mtorr. In order to obtain good thermal equilibrium along the samples in the transport measurements, and to provide good contact between samples and a thermometer, we used sapphire substrates for most samples. For thermoelectric power measurements, however, we used thin cover glasses as substrates, since the good thermal conductivity of a sapphire substrate prevents one from obtaining accurate results. As far as we could judge from x-ray diffraction and resistivity measurements, there is no apparent difference between sapphire and glass substrates.

For the precise measurements of the transport properties, the determination of sample geometry is critical. In order to reduce the error due to different sample geometries, we used thin aluminium masks over the substrates in the sputtering process. These defined samples with five side arms to serve as current and potential leads. The main geometrical error then arises from the determination of the thickness of the samples. To estimate the error in the thickness, we compared the designed layer periodicities with those determined from the x-ray satellite peaks. The agreement was generally good, though we saw up to 10% disagreement in small Λ samples.

The resistivity, the magnetoresistance and the Hall effect were measured by a conventional DC four probe method with Keithley model 181 nanovoltmeters. In order to eliminate thermoelectric voltage and the voltage due to misalignment of contacts, the measurements were repeated with reversed current and field directions. This was done automatically using a microcomputer together with a computer controlled constant current source by way of GPIB. The current used was 5 mA to 20 mA. The magnetic field up to 1 T was supplied by an iron core electromagnet, which could be rotated about a vertical axis, permitting the application of the field in any direction in the horizontal plane. The temperature was measured with a calibrated Au-7% Fe chromel thermocouple.

3. Results and discussion

3.1. Sample quality

The sample quality was checked by x-ray diffraction measurements. As already stated, the Λs determined from the satellite peak positions were generally in agreement with

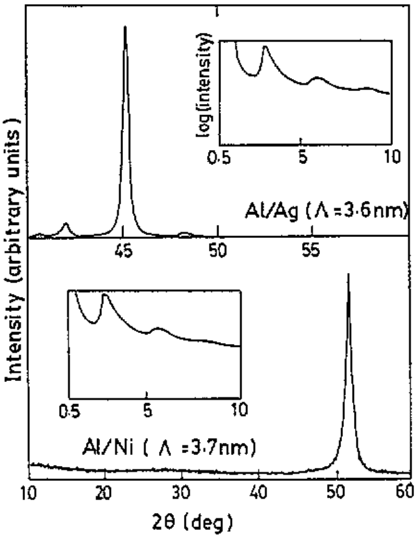

Figure 1. Comparison of x-ray (Co Kα) $\theta - 2\theta$ diffraction patterns for Al/Ag and Al/Ni with similar layer periodicities.

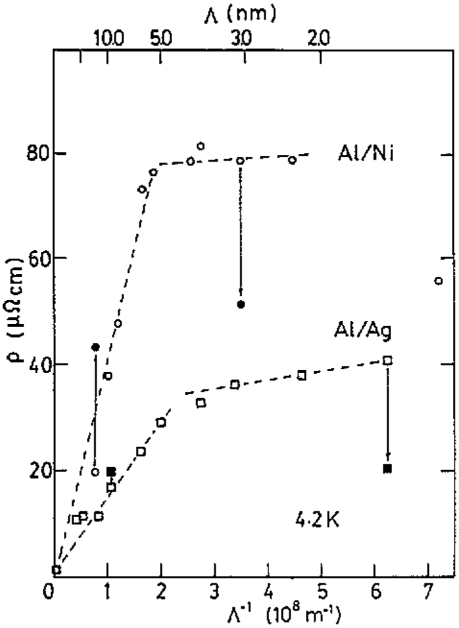

Figure 2. Reciprocal layer periodicity dependence of resistivities at 4.2 K for Al/Ag and Al/Ni. Full symbols are for annealed samples.

the designed values within 10%. As the total thicknesses of the samples we used the values calculated from the x-ray determination, except for the smallest Λ samples and the pure single layer reference samples. Some examples of x-ray $\theta - 2\theta$ diffraction spectra are shown in figure 1. In Al/Ag, both the small angle peaks and the satellites at high angles were observed at least down to $\Lambda = 1.6$ nm, reflecting the layering of the samples. In larger Λ samples, the amplitudes of even number peaks at small angles are reduced compared to the odd ones, confirming the equal thickness of each component. This suggests that possible diffusion and intermetallic compound formation at interfaces are not strong. In Al/Ni, no high angle satellites were observed, while the magnitude of the small angle peaks was essentially the same as those of the Al/Ag system. Since Al and Ag have the same crystal structure and the lattice constants are very close to each other, it is natural to have observed clear high angle superlattice satellites in Al/Ag but not in Al/Ni as discussed by Clemens and Gay [11].

3.2. *Resistivity*

Figure 2 shows the measured resistivities against reciprocal layer periodicity for Al/Ni and Al/Ag. In both systems, the resistivity first increases linearly for large Λ samples and shows a tendency to saturate below about 5 nm. Many multilayer systems show a similar dependence on layer periodicity. The linear dependence can be expected, from the usual size effect theory, due to conduction electron scattering at interfaces [12]. For the limiting values of large Λ, we calculated the resistivity of the parallel combination of the layers. For the bulk resistivity of each element, we used the resistivity measured

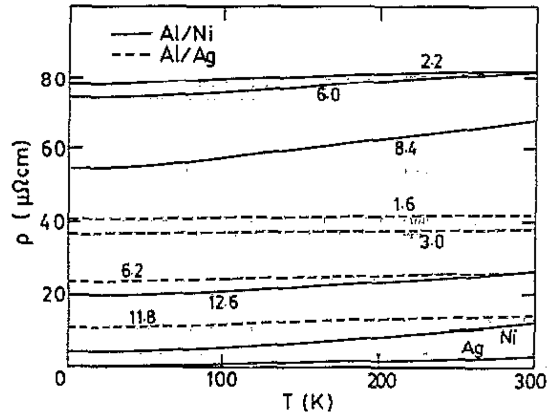

Figure 3. Some examples of temperature dependence of resistivity. The numbers attached to each curve represent the layer periodicities (Λs) of samples in nanometres.

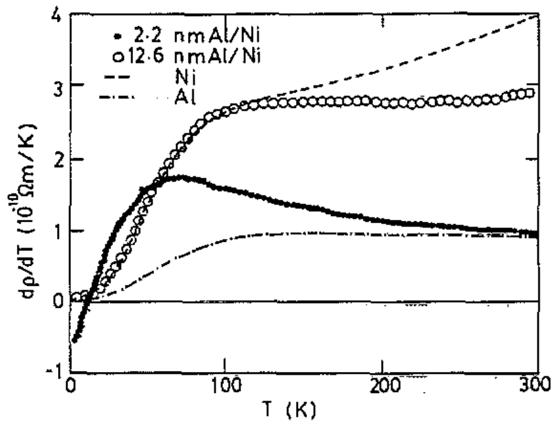

Figure 4. Temperature dependences of the temperature derivatives of resistivity.

on 500 nm single layer samples sputtered in the same manner. As expected, these values lie on the lines extrapolated to the large Λ limit. These facts show that for $\Lambda \leqslant 5$ nm the conduction electron scattering is dominated by that due to interfaces in both systems. To interpret fully the dependence of the resistivity on Λ, the scattering due to grain boundaries should be taken into account, as in the interpretation of the Mo/Al system [13].

Figure 3 shows typical temperature dependences of the resistivity for the two systems. Deviation from Matthiessen's rule are clearly discernible in the Al/Ni system. Such deviations are frequently explained by the competition of different scattering mechanisms with different dependences of relaxation time on wave number. Here it is more likely explained by the different dependence of resistivities of the two elements on layer thickness as in the Gurvich model [14]. Even so, it is not easy to explain the details of the temperature dependence. When Λ becomes smaller than about 5 nm, the resistivity has a minimum near 10 K. The resistivity of the smaller Λ samples shows a clear tendency to saturate above 100 K. This behaviour can be more clearly seen if we plot $\mathrm{d}\rho/\mathrm{d}T$ against temperature as shown in figure 4. In the small Λ samples, $\mathrm{d}\rho/\mathrm{d}t$ is negative at low temperatures and also shows a maximum near 70 K. This behaviour cannot be explained by the simple addition of the contributions from the two elements. The low

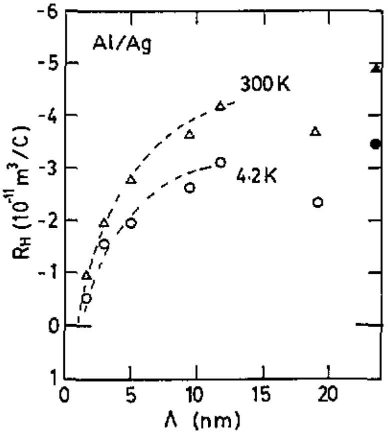

Figure 5. Layer periodicity dependence of the Hall coefficient for the Al/Ag system. Full symbols represent the calculated values from the independent parallel resistor model.

temperature minimum has also been observed on Mo/Ni [15] and Nb/Ni [16] and is thought to be due to the localization effects rather than to the Kondo effect. On a Λ = 2.2 nm sample, we have observed not a negative but a small positive magnetoresistance which is not strongly temperature dependent in the lower temperature region. At present, we cannot give a clear origin of this minimum, but we notice that the low temperature minimum has only been observed in systems containing Ni as one of the components. The resistivity saturation might also be related to some weak localization effect. Several origins for a similar resistivity saturation have been discussed by Allen [17].

3.3. *Hall effect*

The Hall resistivity of Al/Ag system is linearly dependent on the magnetic field for all values of Λ, as expected. The dependence of the Hall coefficient on Λ is shown in figure 5. The Hall coefficient of the large Λ samples is roughly constant and is rather close to that of pure Al. Judging from the x-ray diffraction, no clear indication of intermetallic compound formation is found in the larger Λ samples which have step-like composition modulations. If we assume the parallel resistors model, we can calculate the Hall coefficient of the sample from the resistivities and the Hall coefficients of each component [18]. First, we estimated the resistivity of Al and Ag in each multilayer from the Gurvich model [14]. In the estimate, we used ρl (resistivity times conduction electron mean free path) values of 8.3×10^{-16} Ω m^2 and 6.0×10^{-16} Ω m^2 for Ag and Al respectively [19]. The calculated values at 4.2 K and 300 K are shown at the right hand side of the figure (full symbols). In the limit of large Λ, the experimental values seem to approach the calculated values. In the smaller Λ samples, we observed a clear change of the Hall coefficient in a positive direction. This may be a manifestation of the DC size effect in Ag textures as suggested in [8]. It seems, however, more reasonable to assign its origin to the formation of intermetallic compounds at interfaces. In order to check this possibility, we have investigated the effect of annealing, as will be described later.

The field dependence of the Hall resistivity of Al/Ni systems is shown in figure 6, with Λ as a parameter. For Λ larger than about 6 nm, the field dependence, like that of

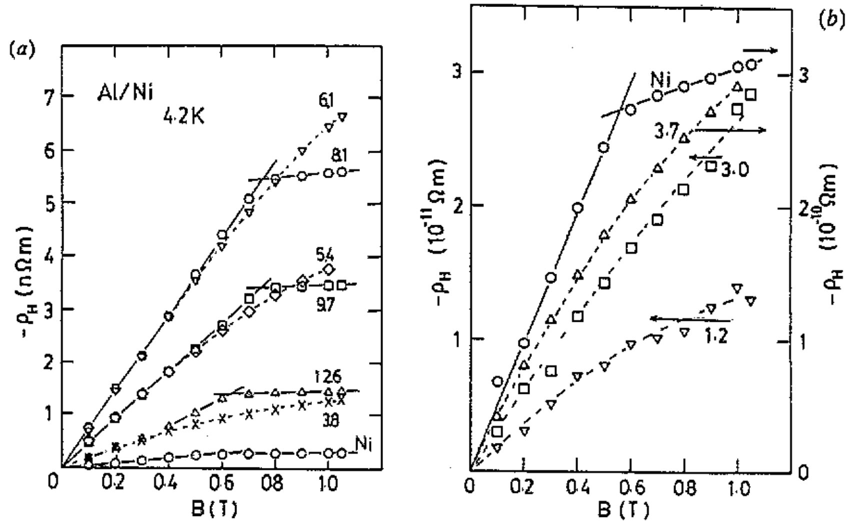

Figure 6. (*a*) and (*b*). Field dependences of the Hall resistivity of Al/Ni system for several values of layer periodicities at 4.2 K.

pure Ni, follows the usual field dependence associated with ferromagnetic materials. This is

$$\rho_H = R_0 H + R_S M$$

where R_0 and R_S are the normal and the anomalous parts of the Hall coefficient and M is the magnetization [20]. From this equation we expect the initial slope of ρ_H to be determined from the combined effects of both the normal and the anomalous parts, while above the saturation field of the magnetization the slope is due only to the normal part. From the break point field we can estimate the saturation magnetization of the samples if there is no contribution from the surface anisotropy. In figure 7 the dependence of the break point field on Λ is plotted together with those reported on Ag/Ni [6]. Since we have not measured the magnetization of this system yet, we assumed a bulk Ni value at least for relatively large Λ samples. In the limit of large Λ, the break point field approaches the bulk Ni value we expected. With decreasing Λ, the break point field increases almost in the same manner as in Ag/Ni down to Λ of about 8 nm. This means that the anisotropy field is negative also in Al/Ni multilayers. The absolute value of the estimated anisotropy field is a little smaller than that of Ag/Ni.

In the smaller Λ samples, we observe no sharp break point field and ρ_H increases smoothly up to 1 T. There are two possibilities to explain this change. The first one is the breakdown of the continuous ferromagnetic film and the resulting formation of small ferromagnetic clusters. If this occurs, we can expect a Brillouin function-like field dependence as was observed experimentally. However, the 3 nm thickness of the Ni layer seems to be too large to expect such breakdown, since we saw very clear small angle satellite peaks in the x-ray experiment. A second possibility is the formation of the intermetallic compound at the interface. It is not easy to identify positively the formation of small amounts of intermetallic compounds because they are generally well textured in such sputtered samples and the expected x-ray peak positions of the textured

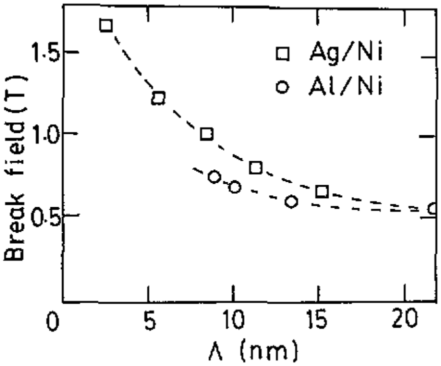

Figure 7. Layer periodicity dependence of the break point field in the Hall resistivity of the Al/Ni system together with the reported result on the Ag/Ni system.

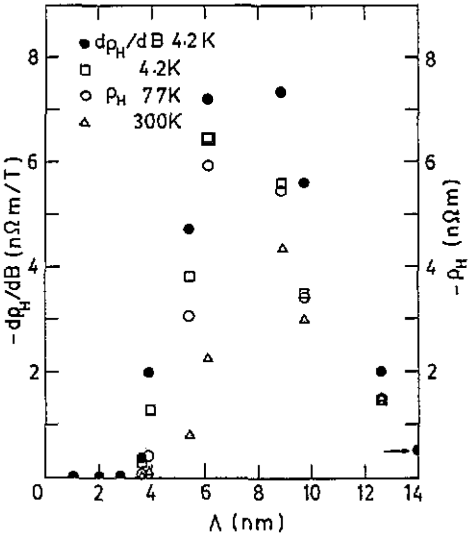

Figure 8. Initial slopes of the field dependence of Hall resistivity at 4.2 K, and the Hall resistivities at 1 T for the Al/Ni system.

Al/Ni intermetallic compounds are close to those of pure Al and Ni. For the Al/Ni system we have observed no ageing effect on x-ray and transport measurements even after keeping the samples one year at room temperature. If intermetallic phases exist, they might be formed only in the sputtering process.

We observed systematic variation of the initial slope of ρ_H against B curves (the anomalous part of the Hall coefficient) as shown in figure 8 along with the Hall resistivity at 1 T. The slope first increases with decreasing Λ down to about 6 nm, where it has a peak, and then decreases to become smaller than that of the 500 nm pure Ni sample below a Λ of about 3.5 nm. These facts show that the magnetic scattering with left–right asymmetry is enhanced with decreasing Λ down to about 6 nm. Combining the resistivity results, we can judge that the interface scattering has such a character. In the smallest Λ sample, the initial slope becomes even smaller than that due to the normal part of pure Ni. A similar behaviour was also observed in Ag/Co and Ag/Ni systems in which we observed a sharp break point field in the ρ_H against field curves down to the smallest Λ sample. In Al/Ni, the absence of a clear break point field below the peak Λ suggests the possibility of intermetallic compound formation at the interfaces.

3.4. *Magnetoresistance*

In the Al/Ag system we only observed a small positive magnetoresistance due to the ordinary cyclotron motion of electrons.

The field dependence of the transverse magnetoresistance at 4.2 K for Al/Ni is shown in figure 9 with Λ as a parameter. The field dependence of the transverse magnetoresistance for systems containing thin Ni films must be interpreted with care. In ordinary ferromagnetic metal films we expect no transverse magnetoresistance anisotropy (whether the field is parallel to or perpendicular to the film) at high fields where

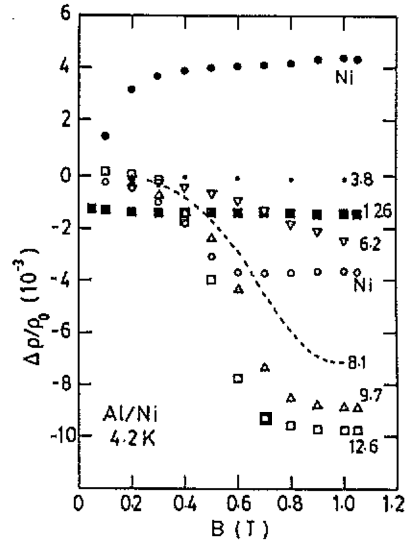

Figure 9. Field dependence of the transverse magnetoresistance at 4.2 K. Open symbols are for the field perpendicular to the layer plane and full symbols are for the field parallel to it.

the magnetization saturates. As was reported in [21], there is a clear anisotropy of the transverse magnetoresistance against the angle between the field direction and the normal to the surface in Ni thin films. For single layer Ni films, the transverse magnetoresistance is positive for the field in the plane and negative for the field perpendicular to the plane. This has been interpreted in [21] as due to the classical size effect. We observed the anisotropy also in Al/Ni multilayers. In figure 9, $\Delta\rho/\rho_0$ for the parallel field are plotted only for selected values of layer periodicity. The magnitude of the negative magnetoresistance for the perpendicular field direction is larger for the $\Lambda =$ 12.6 nm sample and the value, 1%, is larger than that for the 500 nm single layer film but smaller than those of Ag/Ni or Ag/Co (5 to 10%) systems [6].

For the field parallel to the sample plane, $\Delta\rho/\rho_0$ of the 500 nm Ni sample is positive, while for Al/Ni it is negative except for the small Λ samples. The magnetoresistance in a perpendicular field direction tends to saturate at fields close to the break point fields in the Hall resistivity. This means that the field dependence of magnetoresistance in a perpendicular field could also give information about the saturation magnetization or the anisotropy field of multilayer systems. As described above, the transverse magnetoresistance anisotropy is a characteristic feature of Ni films and the existence of the anisotropy is an indirect proof of the existence of a pure Ni thin film in multilayers. At least down to about $\Lambda = 8$ nm, the sharp break point field in the ρ_H against field curve shows that the Ni layer is still continuous. In the smaller Λ samples, we have observed no clear break field, which suggests some breakthrough between adjacent Ni layers. On the other hand, from the magnetoresistance anisotropy, we can conclude that thin Ni layers remain even in the $\Lambda = 3.54$ nm sample. Between $\Lambda = 3.84$ nm and 3.54 nm the low temperature magnetoresistance changes sign from negative to positive. Even if the magnetoresistance becomes positive as a combined effect with the ordinary positive magnetoresistance in the $\Lambda = 3.54$ nm sample, there still remains some sign of the anisotropic magnetoresistance due to thin Ni layers as shown in figure 10. In the $\Lambda =$ 3.0 nm sample there is no detectable anisotropy. This means that the ferromagnetic Ni layer disappears, probably due to the intermetallic compound formation. Of course, we cannot discard the possibility that the thin Ni at the interface becomes a dead layer.

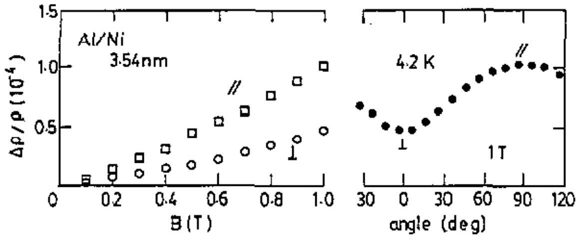

Figure 10. Field dependence and angular dependence of magnetoresistance for Al/Ni with a critical layer periodicity of 3.54 nm at 4.2 K.

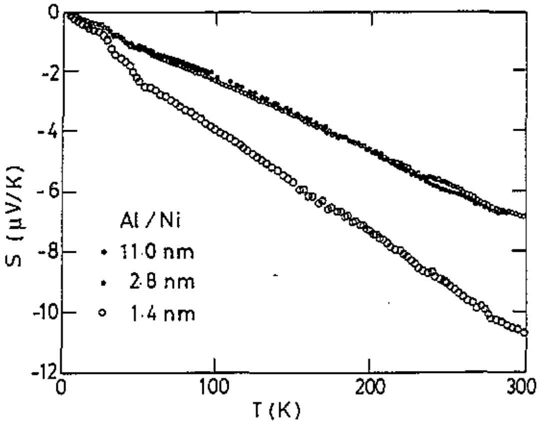

Figure 11. Temperature dependences of the thermoelectric power for the Al/Ni system.

3.5. *Thermoelectric power*

In the Hall effect and the magnetoresistance measurements, Λ was shown to have a critical value of about 6 nm value below which the magnetic conduction electron scattering starts to decrease probably due to the breakdown of continuous magnetic Ni layers. We would also expect some change in the thermoelectric power (S) at this Λ. Surprisingly we observed that the thermoelectric power behaves almost in the same manner above and below the critical layer periodicity as shown in figure 11. Not only the absolute values at 300 K but also the temperature dependence of S for samples with Λ of 11.0 nm and 2.8 nm agree with each other within the experimental accuracy. The absolute value at 300 K is between those of Al ($-1.66\,\mu$V/K) and Ni ($-17.83\,\mu$V/K) [22]. The phonon drag peak reported in pure Al and Ni has not been observed, probably on account of the short electron mean free path in multilayer samples. The value for $\Lambda = 1.4$ nm is clearly enhanced compared to the others and is close to the alloy value [23], which suggests alloy formation at interfaces.

3.6. *Effects of heat treatment*

In Al/Ag, some diffusion or intermetallic compound formation at interfaces occurs slowly even at room temperature, though it is not clearly seen by x-ray diffraction as

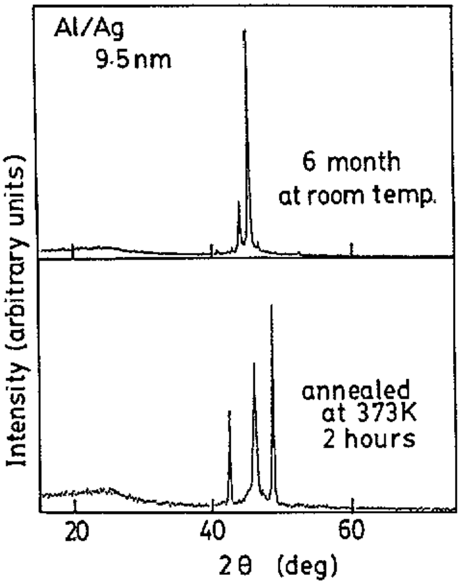

Figure 12. Annealing effect on the x-ray diffraction pattern of the Al/Ag system. The pattern for the as-sputtered stage is omitted, since the only discernible difference from that measured after 6 months is the broad peak near $2\theta = 25$.

shown in figure 12. It becomes evident, however, if we carefully measure the time evolution of their resistivity. In order to see the effect of heat treatment clearly, we annealed two Al/Ag samples at 373 K for 2 hours. The superlattice peaks both at low angle and high angle disappeared and the Bragg peak due to intermetallic phases appeared as shown in figure 12. The transport properties were also affected. The resistance of the larger Λ sample increased while that of the small Λ sample decreased, and they become close to the alloy value reported in [24]. The Hall coefficient of the $\Lambda = 9.7$ nm sample also changes sign from negative to positive and becomes close to the alloy value [24]. At this stage we can infer that the layer periodicity dependence of the Hall coefficient observed on Al/Ag is due to the alloy formation at interfaces.

For the Al/Ni system, the annealing effect on x-ray low angle peaks and on the resistivity is not much different from that of Al/Ag, though the annealing at 573 K is not enough to homogenize the samples completely. The resistivity of the $\Lambda = 3.0$ nm sample is still somewhat larger than that of the $\Lambda = 12.6$ nm sample. The 'reminiscence' of the magnetic components in the Hall effect suggests the existence of some Ni in this annealed sample with $\Lambda = 12.6$ nm, while there is no evidence of a magnetic contribution to the transport properties in an annealed $\Lambda = 3.0$ nm sample [25]. For $\Lambda = 12.6$ nm, the transverse magnetoresistance anisotropy characteristic of a thin Ni film size effect still appears. This means that the magnetic materials in this sample exist as Ni clusters which still have a two-dimensional character. Nastasi *et al* [26] discussed the phase formation at the interface of an Al/Ni bilayer heat treated at 523 K. According to them, electron diffraction patterns taken during the *in situ* annealing of an as-deposited Ni/Al bilayer revealed the presence of $NiAl_3$ after 10 minutes at 523 K, while the induced crystalline component was only AlNi for ion mixed samples. In any case, the possible compounds expected to form at interfaces are non-magnetic. These results suggest that the Λ dependence observed in the Hall effect of Al/Ni cannot be explained only by the alloy formation at interfaces. We must also take into account the loss of magnetism (dead layer) of the thin Ni layer at interfaces.

4. Summary

The existence of x-ray high angle satellites in Al/Ag suggests the possibility of making high quality epitaxial supperlattices, while the alloying at interfaces is inevitable. The formation of small amounts of alloy at interfaces was inferred more easily from the transport measurements than by x-ray analysis.

The Hall effect and the magnetoresistance of Al/Ni show strong Λ dependences. There is a sharp peak between 6 nm and 8 nm in the Λ dependence of the anomalous Hall effect. The peak is explained as the combined effect of the enhancement due to magnetic interface scattering and the reduction due to alloy formation at interfaces. The existence of the transverse magnetoresistance anisotropy confirms the existence of a thin magnetic Ni layer down to a layer periodicity of 3.5 nm.

Acknowledgments

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