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Transport properties of the Cu/Ni multilayer system

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Abstract. The electrical resistivity, Hall effect and magnetoresistance of Cu/Ni sputtered multilayer films were measured between 1.6 and 300 K for a series of different layer periodicities Λ . For small Λ we observed anomalies in the resistivity versus T curves which we interpret as reflecting a reduction in the Curie temperature of these samples. The magnetoresistance at 4.2 K increases with decreasing Λ to about 9.3% for Λ of 2.7 nm compared with 0.3% for a pure Ni film of 500 nm thickness. The anomalous part of the Hall resistivity also increases with decreasing Λ , which suggests that the magnetic interface scattering might enhance the side-jump scattering.

1. Introduction

Recently the galvanomagnetic properties of magnetic multilayer systems have attracted much attention because of the large magnetoresistance (MR) in several systems [1-6]. Together with the experimental studies, many theoretical investigations have been undertaken [7-10]. Several Hall effect measurements have also been reported on magnetic multilayer systems [11-14]. Since the extraordinary Hall effect gives information about the left-right asymmetry of conduction electron scattering, it is useful to measure both the MR and the Hall effect simultaneously on the same samples.

Many physical properties including the transport properties of the Cu/Ni multilayer system have been previously investigated [15–18]. In the course of theoretical investigation of the dependence of MR on transition-metal (TM) elements in Cu/TM multilayers, Inoue *et al* [9] predicted that the MR of Cu/Ni should be greatly suppressed compared with those of Cu/Co and Cu/Fe. There has been, however, no experimental investigation on the systematic study of the MR on Cu/Ni. A Hall effect experiment on Cu/Ni multilayers has already been reported at 300 K [12]. The measurement temperature of 300 K makes it difficult, however, to separate the effect due to the decrease in Curie temperature from that due to the reduction in saturation moment.

In this paper, we report additional results for the electrical resistivity ρ , the MR and the Hall effect of sputtered multilayers of the Cu/Ni system. Measurements were made for a series of layer periodicities A and for temperatures from 1.6 K to 300 K.

2. Experimental procedure

Two series of Cu/Ni multilayers were prepared in an UHV compatible sputtering system (base pressure of about 10^{-9} Torr) at Michigan State University [19]. In series (I), the thicknesses of the Ni and the Cu layers were equal for all samples while, in series (II), only the Cu layer thickness was varied for a constant Ni thickness of 1.4 nm. The total sample thickness was always about 500 nm. To obtain good thermal equilibrium along the samples, and to provide good thermal contact between the sample and a thermometer, polished sapphire substrates were used. During sputtering, we used thin aluminium masks over the substrates in order to obtain a reproducible sample geometry for four-probe measurements.

The resistive and the Hall voltages were measured by a conventional DC four-probe method with Keithley 181 nanovoltmeters. In order to eliminate thermoelectric voltages and the voltage due to misalignment of contacts, measurements were repeated with reversed current and field directions. This was done automatically using a microcomputer-controlled constant-current source with a range form 5 to 50 mA. The temperature was measured with a calibrated Au-0.07%Fe versus chromel thermocouple.

3. Results and discussion

About the magnetization, we refer to the measurement of Wu and Abdul-Razzaq [20] who performed the measurements on samples made in the same system under similar conditions. The sample quality was checked by x-ray diffraction measurements. For multilayer samples, we observed main peaks together with superlattice satellites near the mean position of the (111) lines of Cu and Ni. The spectra are similar to those in [20] for samples from the same source, except that we observed a small satellite peak even for a sample with $\Lambda = 1.4$ nm. This means that the composition modulation remains even in the smallest- Λ sample in the present experiments. The values of Λ determined from the x-ray satellite positions agreed with the nominal values to within 10%. We used x-ray values of Λ to determine the sample thickness needed for the computation of the electrical resistivity and the Hall effect.



Figure 1. The dependence of resistivity on the inverse layer periodicity for series (I) and (II) at 4.2 and 295 K.

3.1. Resistivity

In figure 1 we show the resistivity plotted versus the inverse of the layer periodicity for both series of samples at 4.2 and 295 K. The dependences are nearly linear, although there is some scatter especially in small-A samples. The dependence is different from previous results [12, 18]. Reiss et al [12] find that ρ decreases approximately linearly with Λ^{-1} down to $\Lambda \simeq 2$ nm below which it levels off. The slope of their linear region is about four times larger than the present result, and ρ extrapolated to infinite Λ is about three times larger. The high residual resistivity and the large slope of their results may have resulted from some oxidation of their samples in the sputtering process. The base pressure in their sputtering system was nearly two orders of magnitude higher than that of the present experiment. Sasaki et al [18] obtained magnitudes and the slope of the ρ versus Λ^{-1} curve very close to our results down to Λ of 5 nm, below which, however, they observe a sudden drop in ρ . It is now well known that the layering can be very different in electron beam deposition from that in sputtering, i.e. overall flatness of the interface is usually better in a sputtered sample than in an electron-beam-sputtered sample. One possible explanation of the sudden drop in ρ below 5 nm in electron-beam-grown samples is the short-circuit effect due to the roughness of the interfaces.

The slope for series (II) is larger than for series (I) as expected, since with decreasing Λ^{-1} the relative contribution of the Cu layers (which have smaller ρ) increases. The inverse relationship between ρ and Λ may be interpreted by the classical size effect theory, as was applied to multilayer systems by Carcia and Suna [21]. The present experimental results are, however, not so simple, since one component, namely Ni, has two conductivity channels (spin up and spin down). A model taking into account the two spin channels has been reported by many workers [22], but it is still difficult to apply quantitatively to the present case without adjustable parameters.



Figure 2. Temperature dependence of the resistivity for (a) series (I) and (b) series (II) and 500 nm films of Cu and Ni. A and layer thicknesses are in nanometres.

Here, we discuss only an interesting feature of the Cu/Ni system. The difference between ρ at 4.2 and 295 K increases with decreasing Λ , while it decreases for most previously reported multilayer systems. In order to see the temperature dependence of ρ more precisely, the graphs for $\rho(T)$ and $d\rho(T)/dT$ are shown in figures 2 and 3 respectively. Generally $d\rho/dT$ increases with increasing T below about 80 K, but there is a clear drop above 200 K for the two small- Λ samples. The change in $d\rho/dT$ at about 80 K is due to the transition in the phonon part of both components from a quasi- T^5 to a linear region. We interpret the drop near 200 K to be an indication of the decrease in the Curie temperature in these samples [16], although the possibility of an additional magnetic transition temperature of the type described by Wu and Abdul-Razzaq [20] cannot be ruled out.

This suggests that the increasing difference between $\rho(4.2 \text{ K})$ and $\rho(295 \text{ K})$ with decreasing Λ in figure 1 is related to this reduction in Curie temperature. For Ni-Cu alloys it was reported that an increment in Cu content did not affect the ρ decrease very much below $T_{\rm C}$ but reduced $T_{\rm C}$ itself drastically [23]. This may be related to the effectiveness of the electron-magnon scattering contribution to the resistivity in each channel [24]. On the basis of this [23], we empirically assume that the temperature-dependent part of resistivity can be crudely scaled by a factor of $T/T_{\rm C}$ except at low temperatures. When the fact that the Curie temperature decreases with decreasing Λ in a Cu/Ni multilayer is taken into account [16], the magnetic contribution to the resistivity at 295 K increases with decreasing Λ because of the factor 295 K/T_C. This is at least one reason why the difference between $\rho(4.2 \text{ K})$ and $\rho(295 \text{ K})$ increases with decreasing Λ in figure 1.

3.2. Magnetoresistance

In the present experiment, the MR was always measured in current-in-the-plane-of-layers (CIP) geometry, with the magnetic field both parallel and perpendicular to the film plane. We define the MR as follows:

$$MR = (\rho - \rho_{sat})/\rho_{sat}$$

where ρ_{sat} is the saturation resistivity.



Figure 3. Temperature derivative of resistivity for selected series (I) samples, and 500 nm Cu and Ni films as a function of temperature.

In figures 4(a) and 4(b), we show the results for the MR as a function of magnetic field for Cu(5.6 nm)/Ni(1.4 nm) for *B* parallel and perpendicular, respectively, to the layers. The *B*-parallel results commence with the film as prepared, whereas the *B*-perpendicular measurements were taken after the sample had been saturated parallel to the planes.



Figure 4. CIP MR plotted as a function of magnetic field for Cu(5.6 nm)/Ni(1.4 nm) at 4.2 K for (a) a magnetic field parallel to the sample, and (b) a magnetic field perpendicular to the sample.

This general behaviour, obeyed down to $\Lambda = 1.55$ nm for the Cu/Ni multilayer system, is similar to that exhibited by the Ag/Co and Cu/Co systems and presumably arises from the same origins, i.e. so-called giant magnetoresistance (GMR). According to the theory, the GMR effect may arise when some spin-dependent scattering gives rise to a difference between the resistivities of the two spin bands, i.e. deviation of the anisotropy parameter $\alpha = \rho_L/\rho_{\rm f}$ from 1 is essential. In fact, the clear correlation of the GMR magnitude with the parameter α determined from the deviation from Matthiessen's rule (DMR) in dilute alloys has been reported. For a Cu impurity in Ni, the parameter α has been determined as 2.9-3.7 from DMR measurements, which suggests that the Cu/Ni multilayer is a possible candidate for exhibiting the GMR effect. The field dependence suggests that the interlayer coupling is weakly antiferromagnetic and an antiparallel alignment is realized associated with the domain wall formation [25]. Like the CIP and current-perpendicular-to-the-plane (CCP) results of Pratt et al [26] the initial MR of the virgin samples is higher than that achieved after the sample has been cycled through the saturation field. This would seem to indicate a greater degree of antiferromagnetic alignment in the initial M = 0 state than in the state corresponding to the peak in the MR curve, including the possibility of randomly oriented moments in the individual layers [27].





Figure 5. Resistance of Cu(1.4 nm)/Ni(1.4 nm) as a function of magnetic field direction for B = 0.1 and 1 T. θ is the angle between the magnetic field and the layer plane.

Figure 6. MR ratio with B parallel to the sample surface as a function of Cu layer thickness d_{Cu} for series (I) (O) and series (II) (Δ).

In figure 5 we show, for a Cu(1.4 nm)/Ni(1.4 nm) sample, the MR at 4.2 K as a function of field direction for two fixed-field values of the field magnitude in the transverse geometry. The maximum value of ρ for 0.1 T is approximately the same as that determined from the field dependence for B in plane. The in-plane component of the field at the maximum, 0.1 $T \times \cos(22^\circ) = 0.037$ T, agrees with the field value where a maximum of ρ is observed in the field dependence in-plane. These facts show that the demagnetization field disturbes the magnetic moments to follow the rotation of B up to a certain in-plane component of magnetic field below which the domains are randomized. Above this field, the moments start to align to the field direction within the layer plane.

One important result of the figure is the large MR ratio, although it is smaller than for Cu/Co or Fe/Cr multilayers. In figure 6, we plot the peak MR as a function of Cu layer thickness d_{Cu} for both series of samples, for B in plane. For series (I), the absolute value of the negative MR increases with decreasing d_{Cu} at least down to 1.4 nm where the MR at 4.2 K becomes 9.3% compared with 0.3% for a 500 nm Ni film. The series (II) samples also show a similar increase. In order to see whether there is oscillation in the exchange coupling between neighbouring Ni layers as usually observed for the GMR system, we have also tried to plot the saturation fields of the MR in plane as a function of d_{Cu} (not shown). However, no oscillation has been found within experimental accuracy. This may be because an antiferromagnetic interlayer coupling is small for Cu/Ni as described above. In order to observe the oscillation, if it exists, finer control of the Cu layer thickness is necessary. According to a theoretical study on the material dependence of MR for a (3d TM)/Cu multilayer [9], the magnetic moment and the number of d electrons of the magnetic atoms are shown to be key parameters for the MR, and the MR for Cu/Ni is drastically reduced compared with those for Cu/Co and Cu/Fe multilayers. The large MR ratio in the present experiment suggests that some refinement of the theory is necessary to explain the material dependence of MR.

3.3. Hall effect

The Hall resistivity for Cu/Ni multilayers is shown in figure 7. In the low-field region, both the normal and the anomalous Hall effects contribute to the Hall resistivity, while only the normal part contributes to the high-field slope after the magnetization saturates, i.e.

$$\rho_{\rm H} = R_0 B + R_{\rm S} M \tag{1}$$

where R_0 is the normal Hall coefficient, R_S is the anomalous part and M is the magnetization.

We define the break-point field $B_S(H)$ (H = Hall) as the field where the linear extrapolations of the two regions cross. The meaning of B_S is rather simple, at least for large-A samples, i.e. it is the field to saturate the magnetization of the ferromagnetic layer including the anisotropy field. Wu and Abdul-Razzaq [20] reported unusual magnetic behaviours at low fields in Cu/Ni multilayers resembling spin-glass systems; however, the high-field bahaviour is not very different from ordinary ferromagnetic materials except for small-A samples. For the samples with A < 2.75 nm, the field dependence of R_H is very different and $B_S(H)$ is apparently mugh higher than those for larger A (not shown), which might be related to the unusual magnetic component and should be discussed from a different standpoint.

The A-dependence of $B_S(H)$ for series (I) is plotted in figure 8 together with B_S (MR) (the saturation field for MR with the field perpendicular to the layers). Thus the saturation field values in Cu/Ni estimated from the two transport measurements are reasonably consistent. The results on the Ag/Ni and Al/Ni systems [13] and a 500 nm Ni film are also shown



Figure 7. The field dependence of the Hall resistivity for series (I) at 4.2 K together with a 500 nm Ni film.



Figure 8. A-dependence of the break-point field for series (I) samples estimated from the Hall effect and the MR. Those for the Ag/Ni and Al/Ni are also plotted for comparison.

for comparison. In larger- Λ samples, the Λ -dependences of $B_S(H)$ in the Al/Ni and Ag/Ni systems are very similar [13]. The dependence in the Cu/Ni system is, however, quite different. Even in the $\Lambda = 14.9$ nm sample, $B_S(H)$ is far smaller than that of the pure Ni film, and for $\Lambda > 4$ nm it is weakly dependent on Λ . Perez-Frias and Vicent [28] have reported a similar reduction in $B_S(H)$ for the Nb/Ni system and ascribed this to a dimensional effect in the Ni layers.

In magnetization studies for Cu/Ni with a (111) preferred orientation, Gyorgy *et al* [17] reported that $B_S(M)$ (the saturation field for magnetization with field perpendicular to the plane) increases with decreasing Λ starting from the pure Ni value for the large- Λ limit. Our $B_S(MR)$ and $B_S(H)$ were always smaller than their $B_S(M)$ (e.g. our $B_S(H)$ is about 5 kOe compared with their $B_S(M)$ of about 20 kOe for $\Lambda = 2$ nm, for example) while, for Ag/Co and Ag/Ni, $B_S(MR)$ and $B_S(M)$ agreed. What causes the difference between the Cu/Ni system and the Ag/Co, Ag/Ni and Al/Ni systems? B_S is determined by M_s and the surface anisotropy. The decrease in magnetization by alloying at interfaces does not explain the disagreement, since the reduction in magnetization in the present experiment [20] is not very different from that reported by Gyorgy *et al* [17], and the reduction in M_s was already included in their experimental $B_S(M)$. As suggested by Reiss *et al* [12] for the field dependence of Hall resistivity at room temperature on samples with $\Lambda < 4$ nm, the Langevin-function-like dependence of magnetization due to small Ni clusters in the samples might partly contribute to the difference in B_S ; however, it cannot be a main source, at least for the large- Λ samples.



We next discuss the Λ -dependence of the anomalous Hall component R_S , which is shown in figure 9 together with the normal part of the Hall resistivity (high-field slope). R_S for large- Λ samples is smaller than that for a 500 nm Ni film. With decreasing Λ , it increases and becomes larger than that of the 500 nm Ni sample, while the normal Hall coefficient is almost constant. To understand these dependences, we derived the following approximate formula assuming a parallel-resistor model similar to that applied by Gurvitch [29] to the resistivity:

$$\rho_{\rm H} = \Lambda (\rho_{\rm HA} \rho_{\rm B}^2 d_{\rm A} + \rho_{\rm HB} \rho_{\rm A}^2 d_{\rm B}) / (\rho_{\rm A} d_{\rm B} + \rho_{\rm B} d_{\rm A})^2 \tag{2}$$

where $\rho_{\rm HJ}$ is the Hall resistivity of material J, $\rho_{\rm J}$ the resistivity of the material J in the multilayer and $d_{\rm J}$ is the layer thickness of material J. The formula is basically the same as that derived by Petritz [30] to obtain the mobility and the density of carriers in the space-charge region of a semiconductor surface. Of course, the model is too crude for the detailed discussion of the experimental result; however, we expect that it gives a rough idea of the Λ -dependence of the Hall resistivity of multilayers.

For, Ni/Cu (A \equiv Ni, and B \equiv Cu),

$$\rho_{\rm HA} = R_{\rm 0A}B + R_{\rm SA}M \qquad \rho_{\rm HB} = R_{\rm 0B}B.$$

Inserting these in equation (2), we obtain

$$\rho_{\rm H} = R_{\rm 0M}B + R_{\rm SM}M\tag{3}$$

where

$$R_{0M} = \Lambda (R_{0A} \rho_{\rm B}^2 d_{\rm A} + R_{0B} \rho_{\rm B}^2 d_{\rm B}) / (\rho_{\rm A} d_{\rm B} + \rho_{\rm B} d_{\rm A})^2$$
(4*a*)

and

$$R_{\rm SM} = \Lambda R_{\rm SA} \rho_{\rm B}^2 d_{\rm A} / (\rho_{\rm A} d_{\rm B} + \rho_{\rm B} d_{\rm A})^2. \tag{4b}$$

In equation (3), the first term gives the normal part of Hall resistivity of a multilayer sample. In the present case, R_0 -values for Ni and Cu are not very different. $\rho_{\text{Ni}}/\rho_{\text{Cu}}$ is about 5 in the limit $\Lambda = \infty$, while for finite Λ it approaches 1 because of the size effect from the interface scattering. Putting these values into equation (4), R_{0M} depends only weakly on Λ , from R_0 to at the most $1.4R_0$. This is what we found by experiment within the experimental accuracy, as seen from the data for the normal Hall coefficient in figure 9.

For the anomalous part, we can estimate the A-dependence from equation (4b), if we again apply the simplifying assumption that R_{SA} is the same as that of the 500 nm Ni film. R_{SM} increases from $R_{SA}/18$ to $R_{SA}/2$, as ρ_A/ρ_B varies from 5 (for large Λ) to 1 (for small Λ). The increasing anomalous component with decreasing Λ in the experiment (figure 9) agrees with the estimation; however, the absolute value R_{SM} is unexplainable, i.e. R_{SM} is larger than $R_{SA}/2$ for small- Λ samples. In order to explain the discrepancy, we must take into account the enhancement of the anomalous part in Ni layer due to the increasing conduction electron scattering, as reported for very thin Ni films by Coren and Juretschke [31]. Here we apply the same analysis as was used for the homogeneous ferromagnetic system in order to separate the side-jump and the skew components. A double-logarithmic plot of R_{SM} versus ρ is shown in figure 9(b). For $\Lambda \ge 4.5$ nm, the exponent N in $R_{SM} = a\rho^N$ is close to 2 as expected from the side-jump mechanism. Coren and Juretschke [31] reported similar behaviour in the thickness dependence of the anomalous Hall effect on very thin nickel films. This value is different from $N \simeq 1.5$ determined from the temperature dependence on the 500 nm Ni film.

For series (II), on the other hand, one can assume that R_{SA} is constant for all the samples with the same Ni layer thickness. Then equation (4b) is written as $R_{SM} = R_{SA}(1 + \alpha)/(1 + \alpha\beta)^2$, where $\alpha = d_B/d_A$ and $\beta = \rho_A/\rho_B$. The broken curves in figure 9 were computed for the two constant values of $\beta = 1$ and 5 as examples. No value of β can reproduce the Λ -dependence for series (II) (open triangles in figure 9). As inferred from figure 1, we must take into account the Λ -dependence of the resistivity in Cu layers due to the interface scattering. We write $\beta = \rho_A/\rho_B \propto d_B \propto \alpha$, assuming that ρ_B is proportional to $1/d_B$ owing to the interface scattering for a crude estimation. Then we obtain $R_{SM} = R_{SA}(1 + \alpha)/(1 + C\alpha^2)$, where C is a constant. A fitting curve normalized at Cu/Ni ($\Lambda = 27.5$) is shown in figure 9. As expected, the curve reproduces the experimental data better.



Figure 10. Temperature dependences of the Hall resistivity at 1 T for the smallest- and the largest- Λ samples (series (I)).

We have described above how the reduced Curie temperature of the $\Lambda = 1.55$ nm sample in series (I) affected our results for the temperature dependence of ρ . We naturally expect to see some effect from the same origin on the temperature dependence of the Hall resistivity $\rho_{\rm H}$. The experimental data are shown in figure 10 together with those of the largest- Λ sample. $\rho_{\rm H}$ for the $\Lambda = 14.9$ nm sample increases with increasing temperature as expected from the side-jump mechanism in ferromagnetic materials. $\rho_{\rm H}$ for the $\Lambda = 1.55$ nm sample has a maximum near 160 K above which it decreases drastically. This behaviour is characteristic of the temperature dependence of $\rho_{\rm H}$ near the Curie temperature of ferromagnetic substances [32].



Figure 11. The effect of annealing on the x-ray (Co $K\alpha$) diffraction patterns for the $\Lambda = 14.9$ nm sample.



Figure 12. The comparison of the field dependence of the transverse MR in the field parallel and perpendicular to the plane between the as-sputtered and the annealed states for the $\Lambda = 14.9$ nm sample.

3.4. Effect of annealing

Finally we describe briefly the effect of annealing on the transport properties in order to check the effect of alloying at interfaces on the magnetoresistance. After the $\Lambda = 14.9$ nm sample had been annealed for 15 h at 400 °C, the satellite peaks of the large-angle x-ray spectrum almost fade away as shown in figure 11. The residual resistivity increased about an order of magnitude and is only a little smaller than the homogeneous alloy value. These facts show that the composition modulation was almost destroyed and the sample became a nearly homogeneous alloy film.

The effect of annealing on the negative magnetoresistance is of interest as shown in figure 12. The MR becomes only 0.3% at 1 T in contrast with the 1% MR for a non-annealed sample. The anisotropy due to the demagnetization effect becomes smaller, which suggests the disappearance of the two-dimensional ferromagnetic layers. These indirectly confirm that the enhanced MR with decreasing Λ in Cu/Ni is not due to alloying at the interfaces but is due to the multilayer structure, as in many other magnetic multilayer systems. Another proof of the dominance of the multilayer structure even for small- Λ samples can be also seen



Figure 13. Comparison of the field dependence of the Hall resistivity at 4.2 K and 293 K in the sputtered and annealed states for the $\Lambda = 14.9$ nm sample: --, as-sputtered L = 1.55 nm sample at 4.2 K.

from the annealing effect on Hall resistivity in figure 13. The anomalous Hall resistivity at 4.2 K increases upon annealing in correlation with the increase in the residual resistivity [24]. The decrease in the Hall resistivity at 295 K contrary to the as-sputtered state can be understood as due to the reduction in magnetization. The large difference between the field dependence of the annealed sample and that of as-sputtered 1.55 nm Cu/Ni (broken line in figure 13) also suggests that the multilayer structure still dominates the transport properties even for these small-A samples.

4. Conclusions

(1) The magnitude of the negative MR increases with decreasing Λ down to 2.75 nm where the MR reached about 9.3% at 4.2 K. The origin of the large MR is probably the same as that reported in the other multilayer system, i.e. GMR. We found no clear oscillation of the MR ratio or the saturation field as a function of Cu layer thickness. We believe that the MR maximum as a function of field is ascribed to partial alignment of the magnetizations of neighbouring Ni layers during the magnetization reversal as was suggested for the Ag/Co system [25].

(2) In small-A samples, we have observed, for the first time, the effect of the reduction in the Curie temperature on the temperature dependences of the resistivity as well as the Hall resistivity. We have explained the apparent enhancement of the temperature-dependent part of resistivity with decreasing A in terms of the decreasing Curie temperature. An alternative explanation in terms of a second magnetic transition of the Wu-Abdul-Razzaq type requires further investigation.

(3) The anomalous part of the Hall resistivity also increases with decreasing Λ down to 2.75 nm, suggesting an increasing side-jump component from interface scattering. The Λ -dependence of the break-point field in the field dependence of the Hall effect is different from those reported for Ag/Ni and Al/Ni.

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Acknowledgments

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