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Spin-dependent transport in films composed of Co clusters and C_{60} fullerenes

H. Zare-Kolsaraki[a] and H. Micklitz

II. Physikalisches Institut, Universität zu Köln, Zülpicher Str. 77, 50937 Köln, Germany

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Abstract. Granular films of Co-cluster/C_{60} mixtures have been prepared by the co-deposition of well defined Co clusters (mean diameter ≈4.5 nm) and C_{60} fullerenes onto a cold (≈35 K) substrate. Films having a Co cluster volume fraction $0.23 \le v_{\mathrm{Co}} \le 0.32$ show a resistivity $\rho \propto \exp(T_0/T)^{1/2}$, typical for tunneling with a Coulomb barrier. The tunneling magnetoresistance (*TMR*) has a value of *TMR* $(T \to 0) = 33\%$ for $v_{\mathrm{Co}} = 0.23$ and is decreasing by almost one order of magnitude going to $v_{\mathrm{Co}} = 0.32$. We explain this unusual decrease of the *TMR* with increasing v_{Co} as caused by electron-doping of the C_{60} fullerenes due to the known charge transfer process occurring between transition metal surface and C_{60}. Increasing electron doping may lead to an increasing probability for spin-flip processes within the tunneling barrier, resulting in a decrease of the *TMR*.

PACS. 75.47.-m Magnetotransport phenomena; materials for magnetotransport – 73.40.Gk Tunneling – 73.40.Rw Metal-insulator-metal structures

1 Introduction

Spin-dependent transport in artificially structured, magnetically inhomogeneous systems is an actual field of research. Such artificially structured systems, for example, can be multi-layer systems built of alternative magnetic and non-magnetic layers or granular systems composed of magnetic clusters embedded in non-magnetic matrices. The latter systems nowadays can be produced in a rather well-defined way, namely, by using the co-deposition technique of in-beam prepared metallic clusters together with matrix atoms or molecules onto a cold substrate [1,2]. Various granular systems made of Co clusters embedded in insulating matrices have been prepared with this technique and the tunneling magnetoresistance (*TMR*) of these systems has been studied in the past [3–7]. The *TMR* in these study appeared to be independent of the Co-cluster volume fraction in the insulating matrix [8], i.e. to be independent of sample resistivity, essentially in agreement with the Jullière model [9] wherein the *TMR* is just given by the spin polarization of the *tunneling* electrons. While there is no question about the fact that the electrical transport in these systems occurs by electron tunneling through the rather small matrix molecules (e.g. CH_4, C_2H_4, CO, CO_2, MgF_2), the details of the tunneling process, for example, the character of the tunneling electrons [e.g. Co(4*s*) or Co(3*d*)], is not yet clear [5,7].

A new situation may arise if the Co clusters are embedded in an insulating matrix built from large molecules, e.g. C_{60} fullerenes (so-called "buckyballs"). It is not clear at all if the electrons will tunnel through such large molecules. Furthermore, it is well known that the charge transfer occurring between C_{60} molecules and metal surface [10,11] leads to doped fullerenes which may show metallic conductivity in the case of heavy doping. Therefore, the mechanism of electrical transport in Co-cluster/C_{60} mixtures is an open question. It is for this reason that we have decided to study the electrical transport and especially the magnetoresistance in films composed of Co clusters and C_{60} fullerenes. It should be mentioned that the magnetotransport properties of a very similar system, namely, of a carbon nanotube contacted with ferromagnetic Co electrodes, have been studied recently [12,13]. The observation of a magnetoresistance has been interpreted as evidence for coherent spin transport of the spin-polarized electrons, injected at the Co contacts, through the nanotube having a length of about 250 nm.

2 Experimental set-up and sample preparation

A detailed description of the experimental set-up for sample preparation and magnetoresistance measurements already has been given elsewhere [14]. Granular films are

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[a] e-mail: `kolsarak@ph2.uni-koeln.de`

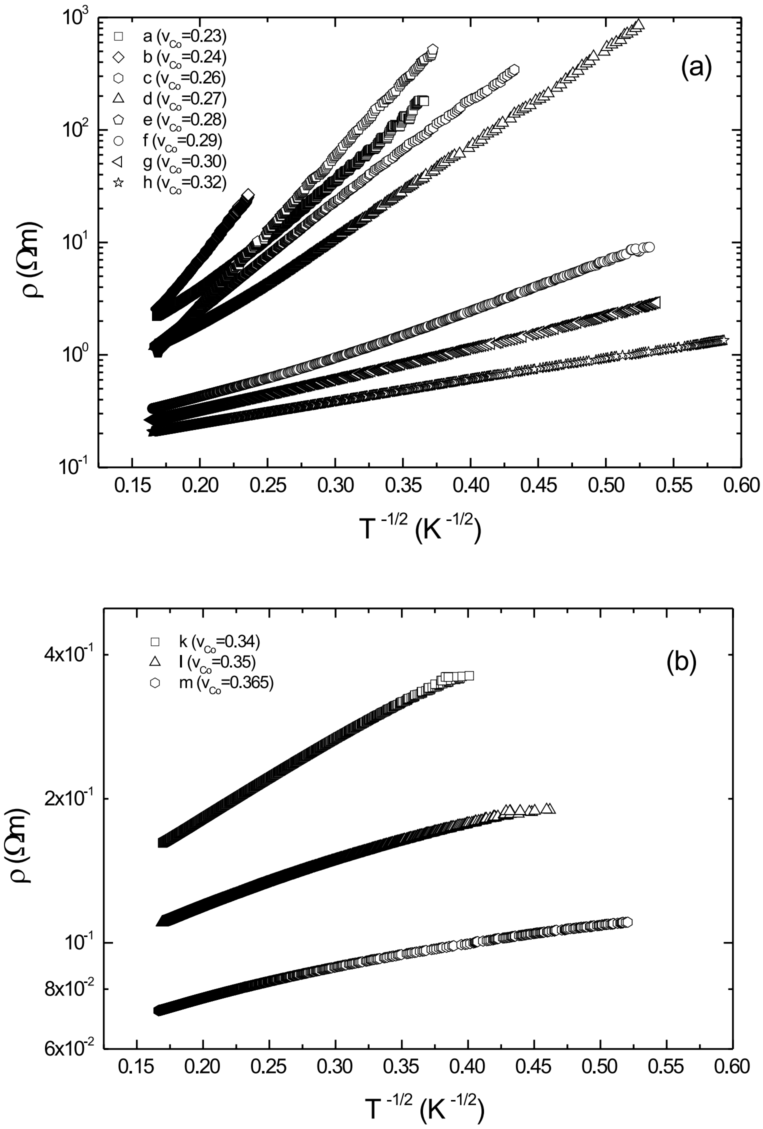

Fig. 1. Resistivity vs. temperature for different *TMR* samples with mean cluster size $L = 4.5$ nm. The linear behavior in (a) corresponds to the expected $\rho \propto \exp(\sqrt{T_0/T})$-law. The samples in (b) do not show such a linear behavior. The Co-cluster volume fraction for different samples is given.

obtained by the co-deposition of in-beam prepared, well-defined Co clusters (mean cluster size ≈4.5 nm) and C_{60} molecules onto a cold sapphire substrate ($T \approx 35$ K) which is mounted onto the coldfinger of a variable temperature ^{4}He cryostat. The ^{4}He cryostat allows measurements in a temperature range of $1.5\ \mathrm{K} \leq T \leq 300$ K. The Co clusters are prepared in an inert-gas (Ar) aggregation cluster source [15]. The C_{60} molecules emerge out of a resistance heated Knudsen cell at a temperature of about 350 °C. Deposition rates of Co clusters and C_{60} molecules are controlled by quartz balances. Typical deposition rates for Co clusters were 1 nm/min. Typical film thickness was 50 nm. The magnetoresistance of the samples has been measured in situ after deposition of the film. Resistance measurements occurred between Ag electrodes which have been evaporated on the sapphire substrate before film deposition and have a length of about 3 mm and a distance of about 2 mm. All resistance measurements were performed by a dc technique using an electrometer which allowed the measurement of resistances up to about 5 GΩ. Magnetoresistance measurements occurred in the sweeping magnetic field ($-1.2\ \mathrm{T} \leq B \leq 1.2$ T) of a split-coil superconducting magnet.

3 Results

3.1 Resistivity

Samples with a Co volume fraction $v_{\mathrm{Co}} \geq 0.23$ (the accuracy of v_{Co} for the samples amounts to 0.01) showed a measurable resistivity ρ with a temperature dependence as shown in Figure 1. Here we have plotted lnρ vs. $T^{-1/2}$ since the resistivity of samples wherein the electronic transport occurs by "variable range tunneling" usually is described by $\rho(T) \propto \exp(T_0/T)^{1/2}$ [16–18]. All samples with $0.23 \leq v_{\mathrm{Co}} \leq 0.32$ essentially show a linear behavior of the lnρ vs. $T^{-1/2}$ curves (see Fig. 1a), i.e. the electronic transport in samples with v_{Co} lying in this Co concentration regime occurs by tunneling processes. The lnρ vs. $T^{-1/2}$ curves are no longer linear for higher Co volume fractions, i.e. for $0.34 \leq v_{\mathrm{Co}} \leq 0.365$, (see Fig. 1b). This indicates that the electronic transport mechanism in these samples has changed. In order to get more information about the transport in our samples we have assumed that $\rho(T)$ for all samples varies as $\rho(T) = \rho_0 \exp(T_0/T)^m$, with an exponent m which may differ from that of "tunneling" samples ($m = 0.5$). In order to obtain the exponent m we have plotted ln$\rho(T)$ vs. T in a double-logarithmic scale and varied ρ_0 until we got straight lines where the slope of these lines gives the exponent m. The result of this procedure is given in Figure 2 where we have plotted the obtained m-values for the different samples as a function of Co volume fraction v_{Co}. The error bars are deduced from the slope of the straight lines in the double logarithmic plots of ρ. All samples with $v_{\mathrm{Co}} \leq 0.32$ have an exponent $m = 0.50(0.10)$ confirming the conclusion we have drawn from the linear behavior seen in Figure 1a. The samples with $v_{\mathrm{Co}} > 0.32$ have an exponent which definitely is smaller than 0.5 and which is decreasing with increasing v_{Co}. Samples with $v_{\mathrm{Co}} \gtrsim 0.50$ essentially show metallic behavior with indication of weak localization, i.e. have a resistivity which slightly increases by about 0.2% going from 35 K to 4 K. Finally we want to mention that the (I, U)-curves of *all* samples show Ohmic behavior up to the highest used voltage of $U \approx 1$ V. This is what one would expect, since the resistance is measured in an in-plane geometry: in this case, the voltage drop at one single tunnel junction between two clusters is of the order $u = U/N \approx 5\ \mu\mathrm{V} \ll k_B T$ (the total number N of the tunnel junctions in series is estimated to be $N \approx b/L \approx 10^5$, with a sample length $b \approx 2$ mm and a cluster distance $L \approx 10$ nm).

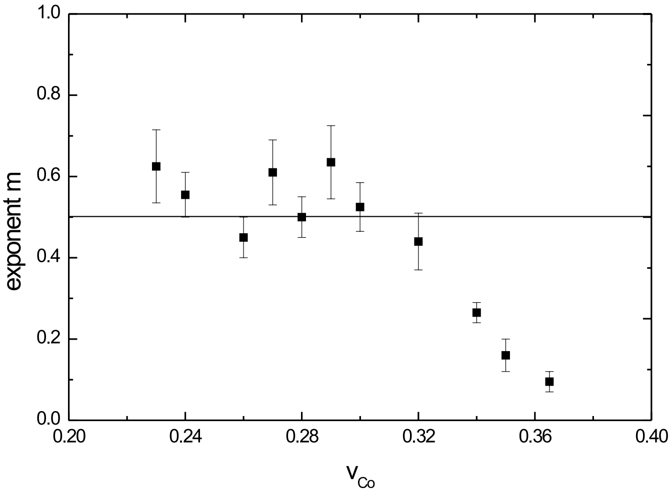

Fig. 2. m-values (ln$\rho = \rho_0 \exp(T_0/T)^m$) for different samples as a function of Co-cluster volume fraction v_{Co}. Samples with $v_{\mathrm{Co}} > 0.32$ have an exponent m which definitely is smaller than 0.5 (see text).

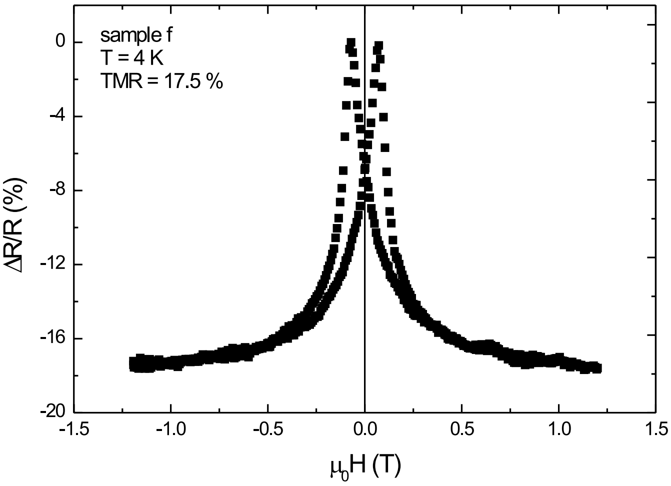

Fig. 3. $\Delta R/R$ as a function of magnetic field $\mu_0\ H$ for sample f at 4 K.

3.2 Magnetoresistance

The resistance R was measured in a sweeping magnetic field at temperatures $T \leq 60$ K. The resistance change ΔR for all samples with $v_{\mathrm{Co}} \leq 0.32$ (samples a-h) showed the well-known hysteretic behavior typical for the tunneling resistance of a granular ferromagnetic systems below its superparamagnetic blocking temperature. Figure 3 gives as an example the relative resistance change $\Delta R/R$ for sample f ($v_{\mathrm{Co}} = 0.29$) having a $TMR = 17.5\%$ at $T = 4$ K. The TMR is defined as $TMR = [R(H_c) - R(H_s)]/R(H_c)$ with H_c and H_s being the coercive and saturation field, respectively. Figure 4 gives the measured TMR values of samples a-h as a function of temperature. The shaded area indicates the region wherein there are all measured TMR values. The dashed line gives the TMR for a system of Co clusters coated with chemisorbed CO molecules and which has the largest TMR values found so far in any granular Co system [7]. The dotted line represents the TMR for Co clusters embedded in rare gas (Kr,Xe) matrices [3]. This TMR seems to be representative for Co clusters embedded in matrices which are not interacting with the Co cluster surface. As it is seen in Figure 4, the TMR values for a given sample decrease with increasing temperature in a way similar to that observed before in all the other granular Co systems we have studied so far [3–7]. The two solid lines in Figure 4 give the fit to the $TMR(T)$ data of samples (a) and (h), respectively, using the model as described in reference [3]. A rather new observation, however, is the following: the TMR at a given temperature strongly decreases with increasing Co volume fraction despite the fact that all these samples still show tunneling behavior in their resistivity (see above). Sample a with $v_{\mathrm{Co}} = 0.23$ has a TMR very similar to that of the Co/CO system [7]. Sample h having the highest Co volume fraction of those samples which still have a resistivity exponent $m \approx 0.5$ (see above) has a TMR value which is

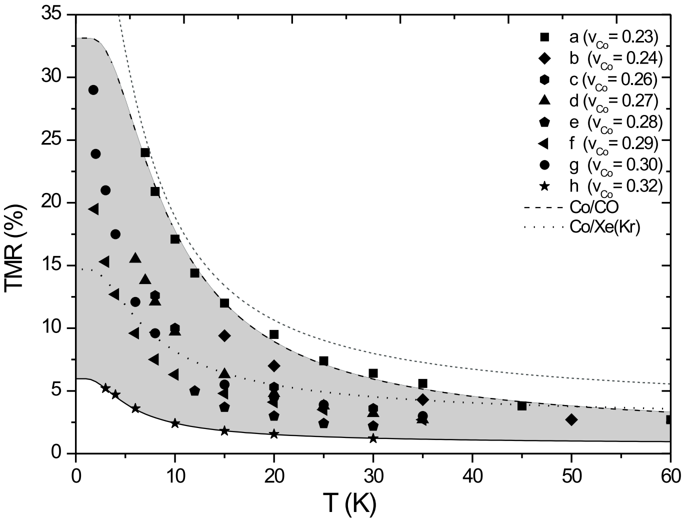

Fig. 4. Temperature dependence of *TMR* for samples a-h. The shaded area indicates the region wherein there are all measured *TMR* values. The dashed line gives the *TMR*(T) for the Co/CO system [7]. The dotted line represents the *TMR*(T) for Co clusters embedded in rare gas (Kr, Xe) matrices [3].

almost one order of magnitude smaller than that of the sample with $v_{\text{Co}} = 0.23$ and also much smaller than that of the system Co/Kr(Xe) [3]. Such a strong decrease in the *TMR* by almost one order of magnitude has *not* been observed before in any of the other granular Co systems we have studied so far [8]. We have plotted in Figure 5 the *TMR* values of all samples showing tunneling behavior in the resistivity ($0.23 \leq v_{\text{Co}} \leq 0.32$) measured at different temperatures and normalized to the *TMR* values of sample a ($v_{\text{Co}} = 0.23$) at the corresponding temperature. One essentially sees a linear decrease in the normalized *TMR* disappearing at $v_{\text{Co}} \approx 0.32$, which is the Co volume fraction where the resistivity exponent m starts to drop below that typical for tunneling samples ($m = 0.5$).

The samples with $0.32 < v_{\text{Co}} < 0.365$, i.e. those samples having a resistivity exponent $m < 0.5$, still show hysteresis in the magnetoresistance indicating some kind of *TMR* but have *TMR* values which are even smaller than that of sample h. All samples with $v_{\text{Co}} \gtrsim 0.50$ have a non-hysteretic magnetoresistance and a $(\Delta R/R)$-value typical for the intrinsic magnetoresistance of metallic Co ($\Delta R/R \approx 0.03\%$ for $\mu_0 H = 1.2$ T) [19].

4 Discussion

4.1 Resistivity

All samples with $0.23 \leq v_{\text{Co}} \leq 0.32$ show a $\ln\rho \propto (T_0/T)^{1/2}$ behavior typical for granular systems where the electrical transport occurs by tunneling with Coulomb blockade. The characteristic temperature T_0 as obtained from the slope of the straight lines of $\ln\rho$ vs. $T^{-1/2}$ plots are lying in the regime 1150 K $\gtrsim T_0 \gtrsim$ 20 K going from $v_{\text{Co}} = 0.23$ to $v_{\text{Co}} = 0.32$. T_0 decreases with increasing v_{Co} as expected, since $T_0 \propto s$ with s the barrier width (see below). An exception is sample e ($v_{\text{Co}} = 0.28$). We do not know why the slope of this sample does not fit to those of the other samples. Apart from that point all data are quite reproducible. The tunneling barrier width s can be estimated from these T_0 values in the following way: $T_0 = 8\kappa s E_c/k_B$ (κ is the wave vector of the tunneling electron and E_c is the Coulomb energy involved in the tunneling process). κ is given as $\kappa = [2m^\star(V_B - E_F)/\hbar^2]^{1/2}$ with V_B being the band gap of the insulating barrier ($V_B = 1.7$ eV) [20] and E_F the Fermi energy of the tunneling electrons. Making the usual assumption that E_F is pinned in the middle of the gap, i.e. $V_B - E_F \approx 1/2V_B$, and assuming an effective mass $m^\star \approx \text{m}_e$ of the tunneling electrons we obtain $\kappa \approx 4.7$ nm^{-1}. Taking $E_c = e^2/2C$ and assuming that the capacitance C essentially is that of a spherical capacitor with an inner diameter given by the mean Co cluster L, an outer diameter of $L + 2s$ and a dielectric constant [21] $\epsilon = \epsilon(\text{C}_{60}) \approx 4.4$ we can estimate s to be $s \approx 0.3$ nm and 0.04 nm for $v_{\text{Co}} = 0.24$ and 0.32, respectively. These s-values definitely are too small since the diameter of a C_{60} molecule sitting between the Co clusters is $D \approx 1$ nm [20]. The s-values obtained from the T_0-values of the *TMR* systems we have studied in the past also have been somewhat smaller than expected from the size D of the molecules sitting between the Co clusters. For the system Co/Kr(Xe), for example, the s-values for the lowest Co cluster volume fractions were about a factor of three smaller than the diameter of the rare gas atoms [3]. We have explained this difference as caused by the formation of percolation aggregates of the Co clusters which leads

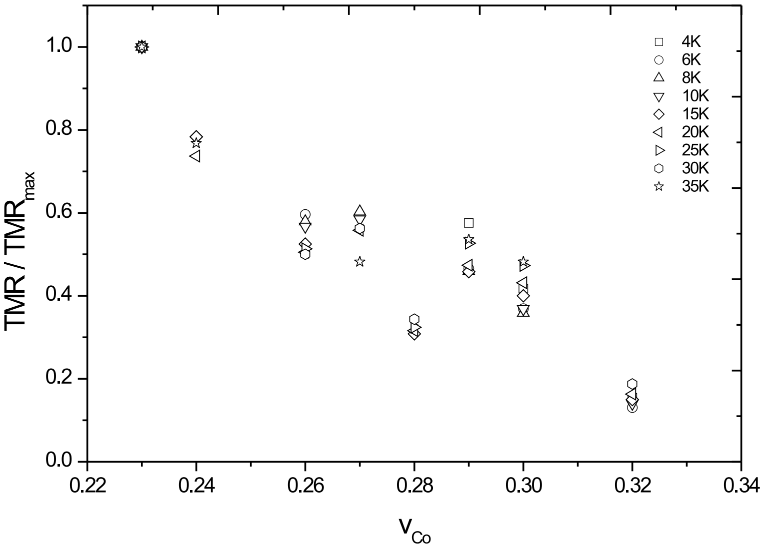

Fig. 5. TMR values of all samples showing tunneling behaviour in the resistivity ($v_{Co} \leq 0.32$) measured at different temperatures and normalized to to *TMR* values of sample a ($v_{Co} = 0.23$) at the corresponding temperature.

to a larger effective Co cluster diameter and thus to a smaller value of E_c. Taking this explanation also for the Co/C_{60} system, we get cluster aggregates of similar size as that in the Co/Kr(Xe) system for small v_{Co}-values, i.e. the ratio of s/D in Co/C_{60} at small Co cluster volume fractions is about the same as that in Co/Kr(Xe). The s/D ratio for samples close to the percolation threshold was about 0.1 for the system Co/Kr while it is about 0.04 for Co/C_{60}. This means that Co cluster aggregation close to the percolation thershold seems to be somewhat larger in the system Co/C_{60} compared to that in Co/Kr. Nevertheless, the overall behavior of the change of s with v_{Co} is not much different in these two systems.

4.2 Magnetoresistance

The most interesting and new result with respect to the magnetoresistance is the observation that the *TMR* in Co/C_{60} mixtures strongly depends on the Co cluster volume fraction v_{Co} (see Fig. 5). As it has been pointed out before, such a change of the *TMR* with v_{Co} was completely absent in all the other systems we have studied before [8]. Let us see if one can understand this change of the *TMR* within the framework of the Jullière model [9].

Within this model the *TMR* is given by $TMR = \alpha P^2/(1 + P^2)$, where P is the spin polarization of the tunneling electrons. The prefactor α takes into account the eventual existence of short range magnetic correlations between the Co clusters. α lies in the range $0 \leq \alpha \leq 2$ going from completely ferromagnetic to completely antiferromagnetic short range correlations. Usually a random distribution of the Co cluster magnetic moments, corresponding to $\alpha = 1$, is assumed. A *TMR* value which strongly decreases with increasing v_{Co} can be obtained if either (i) the prefactor α or (ii) the spin polarization P of the tunneling electrons strongly decreases with v_{Co}. A strong decrease in α with increasing v_{Co} would mean that there are strong ferromagnetic correlations between the Co cluster moments at large v_{Co}-values. We have determined the magnetic hysteresis loops $m(H)$ from the $R(H)$-curves measured at low temperatures (see, for example, Fig. 3) using the relation $[R(H_c)-R(H)]/R(H_c) = P^2m^2(H)/[1+P^2m^2(H)]$ [22]. The analysis of the hysteresis loops resulted in values of the remanent magnetization $m_r = m(0) = 0.5-0.6$ for *all* samples independent of v_{Co}. This is consistent with the assumption of uncorrelated magnetizations, since according to Stoner and Wohlfarth [23] a value of $m_r = 0.5$ is typical for non-interacting single domain particles with a random orientation of their magnetic moments. We, therefore, conclude that $\alpha = 1$ is justified for all samples, i.e. that there are *no* short-range magnetic correlations between the non-aggregated Co clusters.

Next we want to discuss if possible changes in P could be responsible for the observed strong decrease of the *TMR* with increasing v_{Co}. Since P of the individual electrons will not change with v_{Co}, it has to be the *character* of the tunneling electrons which changes with v_{Co}. Such a change in principle is possible since the tunneling barrier width s is decreasing with increasing v_{Co} and the character of the tunneling electrons may change from more s-like to more d-like with decreasing s. The value of the spin-polarization obtained from the $TMR(T = 0)$-value for small v_{Co} or large s, however, is close to the value expected for d-like electrons ($|P| \approx 0.7$, see below) and much larger than that expected for s-like electrons. For that reason the observed strong decrease of the *TMR* with increasing v_{Co} can not be explained in this way. We, thus, come to the conclusion that the only way to get a v_{Co}-dependent *TMR* within the Jullière model is to give up the basic assumption made in this model, namely, that the tunneling process occurs without spin-flip in the tunneling barrier.

If such spin-flip processes occur and in addition the probability for such a spin-flip process increases with increasing v_{Co}, the observed decrease of the *TMR* with increasing v_{Co} could be explained. For that reason we now have to ask the question why there should be spin-flip processes in the tunneling barrier for the system Co/C_{60} but not in the other systems we have studied before and where we did not see such a strong change of the *TMR* with v_{Co}. An answer could be that the well-known electron-doping of C_{60} occurring for C_{60} molecules sitting on transition metal surfaces [10], caused by a charge transfer process between C_{60} and the transition metals, is responsible for these spin-flip processes. Increasing v_{Co} will give increasing electron-doping of C_{60} and, therefore, may result in an increase in the probability for spin-flip processes leading to a decrease of the *TMR*.

Finally we should mention that even if we assume that another transport process than tunneling may be responsible for the electrical transport in mixtures of Co clusters and C_{60} fullerenes we are faced with the problem of a decreasing magnetoresistane with increasing v_{Co}. The only explanation for this experimental fact is to assume that the spin transport is coherent at low v_{Co} and is becoming incoherent with increasing v_{Co}. This explanation should be valid even if the transport occurs not by tunneling but some kind of "hopping with Coulomb gap" [24] which has the same temperature dependence of the resistivity as "variable range tunneling".

4.3 Spin polarization of tunneling electrons

The highest magnetoresistance is observed for sample a ($v_{\mathrm{Co}} = 0.32$), having the lowest Co cluster volume fraction for which a tunneling resistance could be measured (see Fig. 4). Fitting the *TMR* data for this sample with the model as described in reference [3] (the solid line through the data points in Figure 4 is a least-squares fit) we obtain a value of $TMR\ (T = 0) = 33\%$. Using the Jullière model and taking $\alpha = 1$ (see Sect. 4.2) this value of the *TMR* corresponds to a spin polarization P of the tunneling electrons $|P| = 0.7$. This value of P is almost as high as the highest P-value observed until now in a granular Co system, namely that of Co clusters coated with CO molecules which showed a value of $|P| = 0.8$ [7]. The value of $|P|$ for Co/C_{60} as well as that of Co/CO are much higher than that observed for Co clusters embedded in rare gas (Kr, Xe) matrices [3]. In the latter case a $|P|$-value of $|P| = 0.42$ has been found which perfectly agrees with the value determined from the tunneling spectra of ferromagnet-superconductor tunneling junctions [25]. Quite generally it is assumed that the tunneling electrons essentially are s-like electrons. We have explained the high value of $|P|$ observed for Co/CO as caused by the interaction of the CO molecules with the Co cluster surface [7]: hybridization between the Co(3d)- and the C($2p$)-electrons may cause a preferred tunneling of d-like electrons which have a much higher spin polarization than the s-like electrons. Recent ab initio band structure calculations of transition-metal/carbon-nanotube hybrid structures [12] indicate that $|P|$ in these structures can be substantially higher than that calculated for bulk Co. Depending on the detailed atomic structure $|P|$-values between 0.6 and 0.89 are obtained. The $|P|$-value we obtain for Co/C_{60} mixtures agrees quite well with these calculations.

5 Conclusion and outlook

The magnetoresistance in mixtures of Co clusters and C_{60} fullerenes with a low Co-cluster volume fraction ($v_{\mathrm{Co}} = 0.23$) has been found to be (a) strongly enhanced compared to that in the system Co/Kr(Xe) and (b) very similar to that observed, for example, for Co clusters coated with CO molecules. These two experimental facts indicate that it is the interaction of the C atoms with the Co cluster surface which is responsible for the enhancement of the *TMR*. We, therefore, would expect that Co clusters embedded in an amorphous carbon matrix may be another interesting system showing an enhanced *TMR*. Since amorphous carbon does not contain C atoms with double and triple bonds, as they exist in C_{60} and CO, respectively, one also would get information on the possible importance of C double (triple) bonds for the enhancement of the *TMR*. Experiments are in progress in order to examine the *TMR* in this system. The additional observation, namely, the strong reduction of the *TMR* with increasing Co-cluster volume fraction in Co-cluster/C_{60} mixtures is not yet quite understood. Our above given explanation of this reduction as caused by spin-flip processes occurring in the electron-doped tunneling barrier is rather speculative. The above mentioned experiments with Co clusters in amorphous carbon may help to clear up this point since the charge transfer between Co clusters and C atoms in amorphous carbon will not lead to an electron-doped tunneling barrier.

We wont to thank L.H. Tjeng for his suggestion to study the *TMR* in mixtures of Co clusters and C_{60} fullerenes.

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