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Effect of low-temperature ion irradiation on the spin glass *Cu*Mn

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Low-temperature ($T < 10$ K) ion bombardment with He and Ar ions was used to introduce disorder into a $Cu_{97.8}Mn_{2.2}$ thin-film spin glass. In this way the mean free path of the conduction electrons could be varied, and by an *in situ* measurement of the freezing temperature T_f, the corresponding T_f changes were determined. A decrease of T_f was observed for increasing resistivity ρ, exhibiting a linear relation between $\ln T_f$ and ρ. Such a behavior can be interpreted in terms of a damped Ruderman-Kittel-Kasuya-Yosida interaction.

I. INTRODUCTION

Ever since the early measurements of the electrical resistivity behavior of noble-metal-based spin glasses,[1–5] this property has been of interest since it allows one to obtain information about, e.g., the magnetic excitation spectrum. Here, a recent analysis was performed by Campbell *et al.*,[6] who succeeded in describing the temperature dependence of the magnetic contribution to resistivity, $\Delta\rho(T)$, assuming an excitation density of states as calculated by Walker and Walstedt[7] for a Ruderman-Kittel-Kasuya-Yosida (RKKY) spin glass. Excellent agreement is found for temperatures below the freezing-temperature T_f (here T_f is the temperature, where the typical magnetic susceptibility cusp is observed by low-field ac or dc measurements). For temperatures above T_f, where the above description is no longer appropriate, $\Delta\rho(T,c)$ exhibits a broad maximum at a temperature T_m, which depends on the concentration of magnetic impurities c. This behavior has been extensively studied by Larsen,[8–10] who attributes the above maximum to an interplay between the RKKY interaction and a single-impurity Kondo effect. For the latter effect to take over, it is necessary that the long-range interactions are damped out, e.g., by an exponential damping $\simeq\exp(-r/\lambda)$, as suggested by DeGennes for the RKKY interaction[11] [here λ is the mean free path (mfp) of the conduction electrons]. This type of damping, which in case of self-damping leads to a deviation from the scaling behavior $T_f \simeq c$, was also used by Walker and Walstedt[7] and by Kinzel and Fischer.[12] These latter authors concluded that additional nonmagnetic scattering should be most effective in spin glasses with low concentrations of magnetic impurities.

Experimentally, the effect of damping was tested for the archetypical spin glasses by using thin films produced by evaporation into liquid-helium-cooled substrates. The variation of the mfp is then accomplished by a stepwise annealing procedure. For *Au*Fe such experiments were performed by Buchmann *et al.*,[13] who determined T_m as a function of mfp and by Zibold,[14] who measured the mfp dependence of T_f in this way. An enhancement of T_m and T_f was observed by these authors for increasing mfp in qualitative accordance with the theoretical predictions. In particular, the trend concerning additional nonmagnetic scattering as stated by Kinzel and Fischer was confirmed and the usefulness of thin-film spin glasses was demonstrated.

Another experimental approach to vary the mfp in a spin glass was used by Srivastava *et al.*[15] They added nonmagnetic Cu atoms to the spin glasses *Au*Fe and *Au*Mn and determined T_m of the alloys *AuCu*Fe and *AuCu*Mn produced in this way. These authors find good agreement of their $T_m(\lambda)$ results with the Larsen theory.

Recently, the order-disorder transition in Cu_3Au was exploited by Thompson *et al.*[16] to change the mfp in $Cu_3Au(Mn)$ spin glasses, thereby changing T_f. Here T_f was observed to scale like $T_f \simeq \exp(-\langle r \rangle/\lambda)$ for fixed c ($\langle r \rangle$ is the mean interimpurity separation for a given concentration c).

The somewhat controversial experimental results described above seem to justify a totally different experimental approach to test the mfp dependence of T_f. Such a new approach will be reported in the present paper. The basic idea of the method is to start with an annealed thin-film spin glass of *Cu*Mn and to introduce lattice defects into the sample to vary its mfp by low-temperature ($T < 10$ K) ion irradiation. After each irradiation step, T_f is determined *in situ* by measuring the low-field dc susceptibility of the sample with a SQUID (superconducting quantum interference device) system. The low temperatures during the irradiations and measurements are necessary to avoid the annealing of the produced defects. In Cu, the annealing stage I due to the mobility of interstitials starts at approximately 15 K.[17] In thin films ion irradiation allows a large variation of the mfp as compared to corresponding irradiations of bulk samples.[18] This effect is related to the stabilization of defects by impurities present only in films. In the special case of *Cu*Mn thin-film spin glasses, the Mn atoms themselves may be able to stabilize defects leading to a changed local environment, which in turn could influence the magnetic moment. To test this possibility, a study of the irradiation behavior of *Cu*Mn films is necessary. Correspondingly, after a description of the experimental procedure in Sec. II, the paper deals with the resistivity changes of *Cu*Mn films due to low-temperature ion bombardment in Sec. III and with the corresponding changes of the freezing temperature in Sec. IV. The conclusions are drawn in Sec. V.

II. EXPERIMENTAL

A. Sample preparation

A bulk sample of the composition $Cu_{97.8}Mn_{2.2}$ was prepared by comelting high-purity Cu (99.999% purity) and Mn (99.99% purity) under a protective Ar atmosphere. This sample was shaped by cold-rolling, and the final dimensions were $32.0\times3.2\times0.13$ mm^3 with a total mass of 120 mg. After a heat treatment under vacuum at 450 °C for 2 h, the temperature dependence of the dc susceptibility of this sample was measured with a SQUID magnetometer. This magnetometer was especially designed to allow a combination with an ion implantor. In this way the magnetic measurements could be performed *in situ* after low-temperature ion implantation and/or irradiation. Details about this apparatus can be found in Ref. 19. The results obtained for our "mother-alloy" in this way are shown in Fig. 1. Here the magnetization is plotted versus temperature for the following two experimental procedures: In the case of field cooling (given by the closed circles in Fig. 1) the sample is cooled from $T>T_f$ to the lowest temperature of 1.1 K in a magnetic dc field of 4 G and the corresponding data are recorded. In the case of zero-field cooling (open circles in Fig. 1) the sample is cooled from $T>T_f$ to 1.1 K in zero field ($H<0.02$ G), then a field of 4 G is applied and the data are taken for increasing temperatures. The position of the cusp observed in this way defines our freezing temperature T_f. Here a value of $T_f=16.1\pm0.1$ K was obtained and the susceptibility for $T<T_f$ in the case of field cooling was found to be $\chi(T<T_f)=5.3\times10^{-4}$ emu/cm^3. Both values are in good agreement with the results of Nagata *et al.*[20]

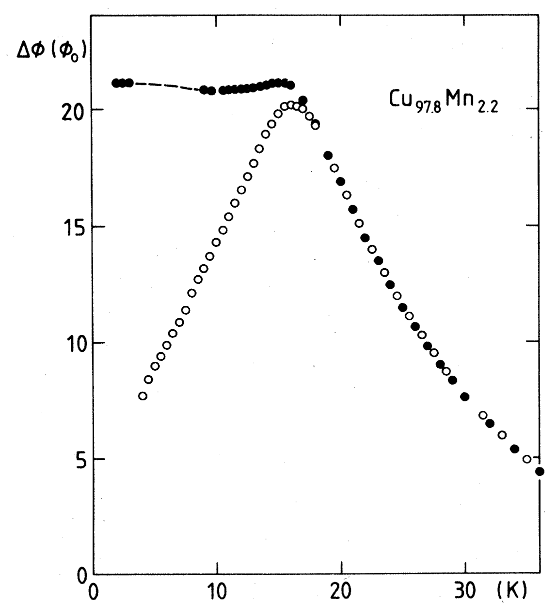

FIG. 1. Magnetic flux changes (in units of a flux quantum ϕ_0) as monitored by the SQUID magnetometer due to the temperature variation of a $Cu_{97.8}Mn_{2.2}$ bulk sample. The closed circles were obtained by field cooling, the open circles by zero-field cooling (details are given in the text).

To produce thin-film spin glasses, pellets from the mother alloy were flash-evaporated at room temperature (RT) onto sapphire substrates. The pressure during evaporation amounted to 5×10^{-6} mbar and the rate was 4 nm/s. All films, which were used for the ion-bombardment experiments, were prepared simultaneously. Optical determination of the film thickness gave $D=220\pm10$ nm. Prior to the ion bombardment, the films were subjected to a further heat treatment under vacuum at 400 °C for 15 min. The resulting electrical resistivity ρ (RT)$=8.15$ $\mu\Omega$ cm is in good agreement with the value found in Ref. 2.

B. Ion bombardments

Generally, two procedures can be distinguished in ion-bombardment experiments, ion irradiation and implantation. Irradiation aims at producing pure radiation damage with a damage distribution as uniform as possible over the film depth. This is accomplished by choosing a projectile energy with a corresponding mean projected range R_p much larger than the film thickness D. In implantation experiments the energy is adjusted to result in $R_p<D$. Thus, atoms are introduced into the sample giving rise to possible chemical effects in addition to the production of damage. If inert gases are used as projectiles as in the following, this chemical effect is thought to be of minor importance.[21] The *Cu*Mn thin-film spin glasses were irradiated with He ions of an energy of $E_0=225$ keV. The corresponding range values are $R_p=630$ nm for the projected range and $\Delta R_p=140$ nm for the range straggling. Thus the irradiation condition $R_p>D$ is very well fulfilled. The ion current density on the target was kept below 1 μA/cm^2, which guarantees that the effective film temperature during bombardment stays below 10 K. Additional Ar bombardments were performed with $E_0=250$ keV. Here the corresponding range values are $R_p=120$ nm and $\Delta R_p=50$ nm, indicating a typical implantation experiment. Again the effective temperature was kept below 10 K. Even if the chemical effect of the implanted Ar is negligible, a problem arises due to the nonuniform damage distribution, which introduces an error in the resistivity determination of the order of 20%. Furthermore, the effect of sputtering, i.e., the thinning of the films by collisions, must be taken into account. For this, a sputtering coefficient of $S=5$ was used.[22] On the other hand, the Ar implantation produces the large number of Cu displacements needed to change the mfp significantly.

Two sets of experiments were performed: First, the effect of ion bombardment on the resistivity ρ of the thin-film *Cu*Mn samples was determined as a function of the ion fluence ϕ, giving $\rho(\phi)$. Then, in a second experiment, films, prepared simultaneously with those used for the first experiment, were bombarded under identical conditions, but now the freezing temperature T_f was measured *in situ*, giving $T_f(\phi)$. Combining the results of both sets of experiments gives $T_f(\rho)$. More experimental details about the implantation setup can be found in Ref. 23.

III. RESULTS AND DISCUSSION

A. Resistivity changes of $Cu_{97.8}Mn_{2.2}$ films due to ion bombardment at $T < 10$ K

1. He irradiation

In Fig. 2 the damage rate $d\Delta\rho/d\phi$ is plotted versus the irradiation-induced resistivity increase $\Delta\rho$ as observed for He irradiation. For small $\Delta\rho$ values ($<1\ \mu\Omega$ cm) a linear decrease of the damage rate is found reflecting an exponential saturation behavior of resistivity as a function of fluence ϕ. Extrapolation to $d\Delta\rho/d\phi=0$ gives a saturation value $\Delta\rho_s^I=1.6\ \mu\Omega$ cm. Added to Fig. 2 is also the corresponding saturation value for pure Cu films (0.3 $\mu\Omega$ cm $\leq \Delta\rho_s \leq 0.4\ \mu\Omega$ cm). Comparing these values one has to conclude that the Mn atoms in the *Cu*Mn films are able to stabilize defects resulting in the higher $\Delta\rho_s^I$ value as compared to pure Cu films. These Mn atoms involved in this stabilization certainly "see" a changed local environment with possible consequences on their magnetic moment.

For $\Delta\rho$ values larger than 1 $\mu\Omega$ cm, clear deviations from the above linear behavior of the damage rate can be seen in Fig. 2. For irradiated metals this is a generally observed effect[17] due to the tendency to form defect clusters. Additionally, in the present experiment on thin films it turns out that also impurities other than Mn atoms contribute to defect stabilization. This effect is more pronounced for Ar bombardments, which will be described in the following.

2. Ar implantation

In Fig. 3 the damage rate curve is presented for the Ar implantation. Again for small $\Delta\rho$ values ($\Delta\rho < 1.5\ \mu\Omega$ cm) corresponding to small Ar fluences ($\phi < 6\times10^{13}$ cm^{-2}) a linear behavior is observed as for the He irradiation. The fact that the damage rate for Ar bombardment is numerically much larger than for He irradiation reflects the higher nuclear energy loss of the heavier projectile. But extrapolation of the linear part in Fig. 3 results in the same saturation value of $\Delta\rho_s^I=1.6\ \mu\Omega$ cm as obtained by He irradiation. On the other hand, this value can be enlarged by approximately an order of magnitude by bombarding with high Ar fluences. Comparison to previous results on Al, In, Pd, Zn, and Pb films[18] suggests that the high $\Delta\rho$ values observed after bombardments with Ar fluences $\phi > 10^{15}$ cm^{-2} must be attributed to impurity-stabilized defect complexes. Here the impurities, especially oxygen, are introduced into the films by the irradiation process itself either by ion-beam mixing from the substrate or by internal recoil implantation from, e.g., oxide precipitates. The detailed structure of these defect complexes is not known, but if a physical property like T_f is depending on a "global" parameter like the mfp, such detailed knowledge is not necessary. In this case, the creation of defect complexes is just one way to significantly decrease the mfp.

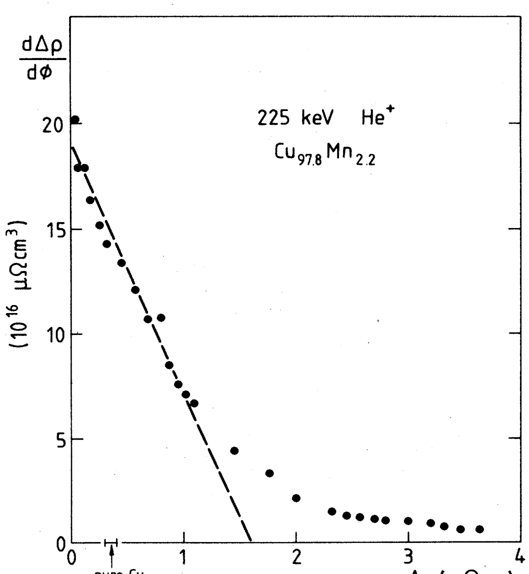

FIG. 2. Damage rate $d\Delta\rho/d\Phi$ produced in a $Cu_{97.8}Mn_{2.2}$ thin-film spin glass by low-temperature ($T<10$ K) He bombardment as a function of the corresponding resistivity increase $\Delta\rho$ (Φ stands for the He fluence). The linear part is indicated by the dashed line.

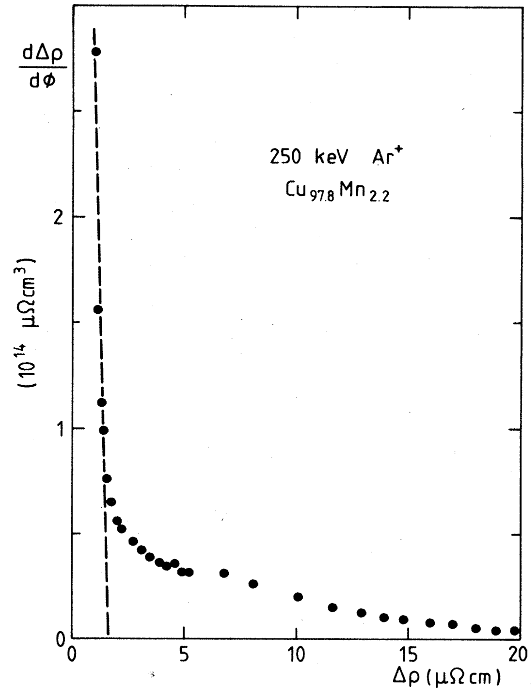

FIG. 3. Damage rate $d\Delta\rho/d\Phi$ produced in a $Cu_{97.8}Mn_{2.2}$ thin-film spin glass by low-temperature ($T<10$ K) Ar bombardment as a function of the corresponding resistivity increase $\Delta\rho$. The linear part is indicated by the dashed line.

B. T_f changes due to ion bombardment of $Cu_{97.8}Mn_{2.2}$ films at $T<10$ K

As described in Sec. II, in a second set of experiments the effect of low-temperature ion bombardment on the freezing temperature was studied. In Fig. 4 typical results of such an experiment are shown. Here the magnetic sus-

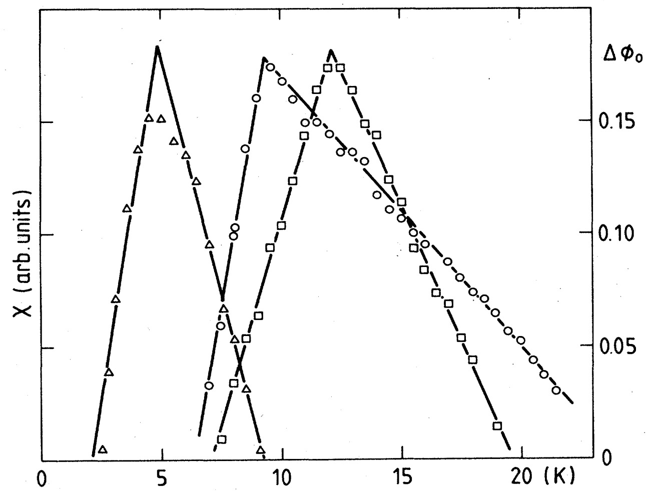

FIG. 4. Magnetic susceptibility (left scale, arbitrary units) and magnetic flux changes (right scale in units of a flux quantum Φ_0) of a $Cu_{97.8}Mn_{2.2}$ thin-film spin glass versus temperature as obtained by zero-field cooling. Open squares: prior to ion bombardment, ion fluence $\Phi=0$. Open circles: after He irradiation with $\Phi_{He}=2\times10^{16}$ cm^{-2}. Triangles: after additional Ar bombardment with $\Phi_{Ar}=10^{15}$ cm^{-2}. From the intersection of the straight lines the freezing temperature was determined.

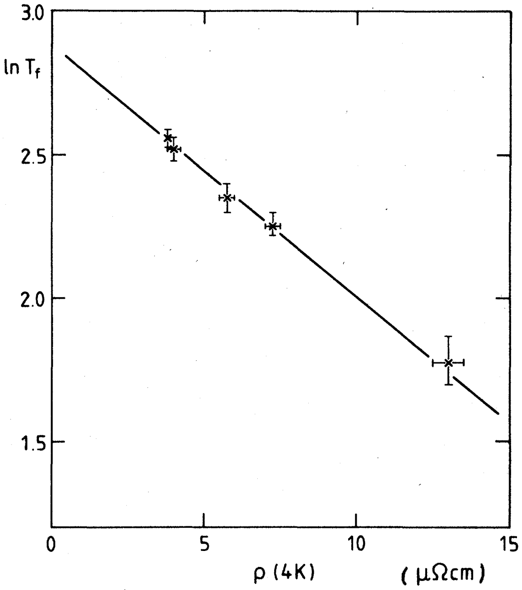

FIG. 5. Logarithmic plot of the spin-glass freezing temperature T_f as a function of the residual resistivity ρ. The solid line is obtained by a least-squares fit to the data.

ceptibility (left scale, arbitrary units) and the number of the flux quanta $\phi_0=h/2e$ as monitored by the SQUID (right scale) are plotted versus temperature for a $Cu_{97.8}Mn_{2.2}$ film subjected to different ion bombardments at $T<10$ K: The open squares give the result prior to ion bombardment ($\phi=0$), the open circles were observed after He irradiation with $\phi=2\times10^{16}$ cm^{-2} and the triangles were found after additional Ar implantation with $\phi=10^{15}$ cm^{-2}. Intersecting straight lines are fitted to the data as shown in the figure and from the corresponding intersection T_f is deduced. Clearly T_f is decreased by the ion bombardment. It should be noted that curves like those presented in Fig. 4 are not unequivocal, but depend on the details of the cooling procedure even if zero-field cooling is performed. Thus, no quantitative conclusions can be drawn from the absolute values of $\Delta\phi_0$. But in any case, a clear kink of the curves is observed allowing to determine T_f. In detail, each $\Delta\phi_o$-T curve was measured three times and the corresponding T_f values were averaged. A typical uncertainty of T_f found in this way is ±0.5 K.

In Fig. 5 our main result is presented. For this figure the $\rho(\phi)$ and the $T_f(\phi)$ results are combined to give a plot of $\ln T_f$ versus resistivity ρ. The data can be very well described by a linear behavior as shown by the solid line in Fig. 5, which exhibits a slope of $d\ln T_f/d\rho=-0.086$ $\mu\Omega\,\text{cm}^{-1}$. But the limited range of ρ values does not allow one to extrapolate to $\rho\rightarrow\infty$, in order to check whether or not a finite T_f is approached in this limit.[24] The results suggest the correlation $T_f\simeq\exp(-\langle r\rangle/\lambda)$ as found in Ref. 16. Here the average interimpurity distance corresponding to $C_{Mn}=2.2$ at. % is $12.3\,\text{Å}$. Taking this value for $\langle r\rangle$ and assuming $A=\rho\cdot\lambda$, from the above slope a value of $A=1.43\times10^{-12}$ $\Omega\,\text{cm}^2$ is deduced. The value of A is smaller by a factor 4.5 than, e.g., the free-electron value for Cu, $A=6.42\times10^{-12}$ $\Omega\,\text{cm}^2$. This difference can be expressed as an effective mfp for the RKKY interaction as opposed to a transport mfp. In the Larsen theory[10] this effect is accounted for by introducing a parameter β, which in our case, should be equal to 4.5, a value acceptable for a three-dimensional impurity. On the other hand, a quantitative application of the Larsen theory demands an unphysically large β value of about 14 to fit the data; this theory gives much too small a value for the T_f decrease. This is most easily seen by calculating the initial T_f depression $(d\ln T_f/d\rho)_{r\rightarrow0}$ according to the Larsen theory (r is the dimensionless damping parameter as defined in Ref. 10). With $\beta=4.5$, $\rho=4$ $\mu\Omega\,\text{cm}$, $c=0.022$ (Mn concentration), $a=3.61\times10^{-8}$ cm (lattice constant for pure Cu), one obtains $(d\ln T_f/d\rho)_{r=0}=-0.03$ $(\mu\Omega\,\text{cm})^{-1}$ as compared to the experimental value of -0.086 $(\mu\Omega\,\text{cm})^{-1}$. It should be noted that to apply Larsen's formula for the above limit, it must be guaranteed that $rc\ll1$. In our case with the above values, one has $rc=0.08$, i.e., reasonable fulfillment of the rc requirement. Thus, the Larsen theory fails to predict the T_f depression quantitatively, giving too small values. Such a trend was also found in Refs. 13 and 14. The simplistic approach $T_f\simeq\exp(-\langle r\rangle/\lambda)$ accounts for the results, but for λ a value smaller by a factor of 4.5 must be used as compared to transport the mfp. This is in agreement with the arguments given by Larsen[10] and the findings of Walker and Walstedt.[7,25]

Though in accordance with the damping idea, the above results are somewhat surprising, especially since recent work indicates that the RKKY interaction is too simple an approach for *Cu*Mn spin glasses.[26] Therefore, some processes, which are characteristic for ion irradiation and which may be important in the context of T_f, should be mentioned.

(a) The results of Sec. III A clearly indicate that a part of the Mn atoms stabilize defects, resulting in a changed local environment with possible consequences on the magnetic moment.

(b) Due to collisional processes Mn atoms may end up at interstitial sites and at low temperatures they can remain there, again resulting in a new local environment.

(c) From previous experiments[18] it is known that oxygen is present in thin films mainly in the form of oxides precipitated at grain boundaries. By collisions, oxygen can be redistributed and a finite chance exists that it comes to rest near a Mn atom, again influencing the magnetic moment. A further process is ion beam mixing at the interface substrate/film resulting in a transport of, e.g., oxygen from the substrate into the film.

(d) Similarly to (b), collisional processes can change the Mn short-range order, which is known to exist in *Cu*Mn samples.[27]

(e) There could be an effect of implanted projectiles (He,Ar) on T_f.

Though all of these effects could influence T_f, some of them appear to be negligible in the case of He irradiation. This especially holds for process (e). Here, under given irradiation conditions, a He fluence of $\phi=1.5\times10^{15}$ cm^{-2} corresponds to a concentration of implanted species of the order of 1 ppm. It is hard to believe that this small concentration is responsible for the 30% reduction of T_f observed at this fluence. Also all processes which demand a mass transport due to collisions like the redistribution of manganese or oxygen atoms are of minor importance for the He irradiation. The reason is that these processes are proportional to the nuclear energy loss $(dE/dx)_n$, which is very small in the He case. The following estimate may illustrate this: Assuming a typical displacement threshold energy of $E_d=25$ eV and an average nuclear energy loss of 0.35 eV/A for 225 keV He ions in Cu, one obtains for the average number of displacements per Cu atom after irradiation with $\phi_{\rm He}=1.5\times10^{15}$ cm^{-2} the value 0.01, and only 2% of these displacements involve a Mn atom. Thus, only process (a) could be responsible for the observed T_f decrease if the RKKY approach is thought to be too simplistic for *Cu*Mn.

In this context, it is important to note that the result for the Ar implantation (data point at 13 $\mu\Omega$ cm in Fig. 5), where the processes (b)—(e) certainly play a crucial role, seems to be a continuous extension of the He results as far as T_f is concerned. Since for this type of bombardment the transport of atoms from the substrate and from grain boundaries becomes important as a source of defect-stabilizing impurities, one has to expect a large variety of different impurity-defect complexes rather than well-defined defects like, e.g., interstitials and vacancies. Taking this into consideration leads to the conclusion that the T_f decrease can be described by a "global" parameter λ and is not due to a specific type of defect. But again this does not rule out the possible effect of the local environment, which can be changed by defects on the magnetic moment and thus, on T_f.

IV. CONCLUSION

Low-temperature ion bombardment of a $Cu_{97.8}Mn_{2.2}$-spin glass combined with *in situ* T_f measurements has been used for the first time to produce changes of the mfp and to study the corresponding effect on T_f. This experimental approach proved to be useful. A decrease of the freezing temperature was found for decreasing mfp's, indicating that the Mn-spin interactions are dominantly conduction-electron mediated. Quantitatively, a linear relation between $\ln T_f$ and the resistivity ρ was observed. Such a behavior is most easily described by an exponentially damped RKKY interaction. If interpreted in this way, a mfp must be assumed, which is by 4.5 smaller than the transport mfp. The slope of the T_f depression is larger than what is calculated from the Larsen theory. By combining He and Ar bombardments, where different types of defects are created, one concludes that the T_f behavior is not due to a specific defect but rather to a general mfp effect independent of the detailed defect configurations. The study of the damage rate for these bombardments gave clear evidence that Mn atoms are stabilizing defects. A specific influence of the corresponding changed local environment on T_f cannot be excluded.

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