Effect of aging heat treatment on the magnetic behavior of a gold-iron alloy

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(Received 18 April 1983)

Susceptibility and magnetization measurements at temperatures below 200 K indicate the absence of long-range ferromagnetic spin order in $Au_{82}Fe_{18}$, aged at 200 °C, in spite of a considerable increase of the internal susceptibility as a result of the aging treatment.

From their recent x-ray diffuse scattering study of Au-Fe alloys with 14.4, 15.4, and 19 at.% Fe, Dartige, Bouchiat, and Monod[1] concluded that in these alloys essentially all Fe atoms are segregated in small platelike regions (Guinier-Preston "zones") parallel to {210}-type planes. These platelets were found to be only about two atomic layers thick, and they extend, on the average, approximately 30 Å in directions parallel to their plane. From these dimensions one can calculate that the number of atoms per platelet is, on the average, about 120. Assuming that all these atoms are Fe, and that the ferromagnetic spin alignment within such a cluster of Fe atoms is complete, the cluster moment associated with a platelet would be about $264\mu_B$, with $2.2\mu_B$ per Fe atom. If an Fe moment of $2.35\mu_B$ is assumed in such a gold-rich nearest-neighbor environment, as derived from the saturated spontaneous magnetization at 1.6 K (Ref. 2), the cluster moment would be about $282\mu_B$. On the other hand, the "average" magnetic cluster moment for $Au_{82}Fe_{18}$ in the quenched state, derived earlier from magnetization σ versus field H isotherms, measured between 70 and 170 K in 10 K intervals, was found[2] to increase from about $30\mu_B$ at the highest temperature to $180\mu_B$ at 75 K. While these values are *lower* than those estimated above on the basis of the x-ray diffraction results,[1] they are consistent with the latter if the assumption of perfect ferromagnetic alignment of all spins within a platelet is dropped. Certainly nothing here indicates that the cluster moment values derived from magnetization data by least-squares fitting of a Brillouin function, modified to take into account the interaction of the cluster moments with their magnetic environment,[2] are "unphysically large," as suggested by Dartige, Bouchiat, and Monod.[1] In fact, the x-ray diffuse scattering results,[1] showing the existence of Fe atomic clusters, provide evidence confirming the treatment of these "spin-glass" alloys in terms of magnetic clusters, as was done in Refs. 2 and 3.

On the basis of a similar analysis of $\sigma(H)$ isotherms for $Au_{85}Fe_{15}$ (Ref. 3), it was concluded[2,3] that in the quenched state 64—73 % of the Fe moments in this alloy are participating in magnetic clusters. Scheuer, Loewenhaupt, and Just[4] demonstrated in a diffuse magnetic neutron scattering study of $Au_{85.5}Fe_{14.5}$ that the quasielastic part of the energy spectrum of the magnetically scattered neutrons consisted of two Lorentzian lines of different widths, corresponding to fast relaxation (attributed to single Fe moments) and slower relaxation (attributed to Fe moments associated with clusters). By using the ratio of the areas under the two Lorentzian curves,[4] I note that approximately 72% of the total quasielastically scattered neutron intensity is due to Fe moments in clusters, in excellent agreement with the above-mentioned analysis[2,3] based on magnetization data for $Au_{85}Fe_{15}$.[3] On the other hand, the magnetization data for quenched $Au_{82}Fe_{18}$ show[2] that in this alloy essentially all Fe moments are participating in magnetic clusters, in agreement with the estimate of Dartige, Bouchiat, and Monod.[1] One may therefore expect that a neutron magnetic diffuse scattering study similar to the one in Ref. 4 would find for $Au_{82}Fe_{18}$ only *one* Lorentzian line, corresponding to the slower relaxation characteristics of Fe moments in magnetic clusters.

Since each of the very large number of tiny iron-rich platelets in the 15 and 18 at.% Fe alloys becomes ferromagnetic upon cooling below their various Curie temperatures, it is understandable that neutron scattering studies[5,6] at low temperatures indicate ferromagnetic spin correlations in alloys in the above composition range. Also, Mössbauer polarization studies of similar Au-Fe alloys show ferromagnetic hyperfine splitting at low temperatures.[7,8] Both types of measurement mentioned are reacting to *short-range* ferromagnetic spin order. The range of Curie temperatures of the platelets, as shown by both the magnetization data[2,3] and the temperature dependence of the hyperfine splitting,[7,8] is largely between 90 and 115 K in quenched $Au_{85}Fe_{15}$ and between about 160 and 175 K in quenched $Au_{82}Fe_{18}$, i.e., very far below the Curie temperature of bcc α-Fe. This great decrease of the Curie temperature may be partly due to the coherency of the Fe platelets with the Au lattice, causing them to retain the fcc structure. But clearly also important in this respect is the very small size of the platelets. In fact, considerable increase in the high-temperature end of the range of cluster Curie temperatures was observed in Au-Fe alloys by means of both Mössbauer hyperfine splitting studies[9] and of alternating field susceptibility measurements[10] as a result of aging heat treatments at temperatures beween about 200 and 383 °C, which may allow the platelets to grow larger.

In spite of the presence of very many small ferromagnetic particles (occupying a total of about 18% of its volume), the quenched alloy $Au_{82}Fe_{18}$ as a whole does not develop long-range ferromagnetic spin order at any temperature. This is also indicated, in addition to the arguments presented earlier,[2] by the maximum value of its sus-

ceptibility. It was found[2] that a spherical quenched specimen of this alloy below about 150 K has an internal susceptibility χ_i much larger than its reciprocal demagnetization factor, so that the external susceptibility $\chi_e=(\chi_i^{-1}+D)^{-1}$ reaches a plateau of $\chi_e=\sigma/H_a=1/D$, where the demagnetization factor is $D=(4\pi/3)\rho$ for a spherical specimen of density ρ(g/cm^3). Using a lattice parameter value of $a_0=4.021$ Å (at 300 K), obtained by interpolation from (Ref. 11), one gets $\rho=17.44$ (g/cm Oe) and $1/D=0.0137$ emu/g. As seen in Fig. 1 of Ref. 3, the plateau of the measured external susceptibility for a spherical specimen of $Au_{82}Fe_{18}$ in the quenched condition agrees with this value well within the experimental error. For a thin-sheet quenched specimen of the same alloy (for which D is much smaller than for a sphere, provided that the field is applied parallel to the plane of the sheet) the limiting value of χ_e is correspondingly larger. As seen in Fig. 4, Ref. 2, in this configuration the external susceptibility of the same alloy reaches a maximum value of $\sim 2.5(10^{-2}$ emu/g Oe) and it does not have a well-defined plateau. The curve is rather well rounded at the top, and the descending temperature curve lies much higher than the ascending branch since the external susceptibility is not effectively limited here by the reciprocal demagnetization factor. Consequently, the maximum of the internal susceptibility is of the same order of magnitude as that of the measured external susceptibility, i.e., $\sim 2.5(10^{-2}$ emu/g Oe), which is far below the susceptibility of a ferromagnet.

The finding that quenched $Au_{82}Fe_{18}$ has no long-range ferromagnetism at any temperature is consistent with the strong negative magnetoresistance found[12] in quenched $Au_{81}Fe_{19}$ in the entire temperature range below ~ 200 K. The maximum of the negative magnetoresistance at ~ 170 K is presumably due to the maximum (but not infinite) value of the internal susceptibility of this mictomagnetic alloy, having short-range ferromagnetic spin order. In Ref. 12 the term "quasiferromagnet" is used for such alloys.

Crane and Claus reported recently[13] that $Au_{86}Fe_{14}$ becomes ferromagnetic after heat treatments, which involve quenching a small specimen from ~ 950°C and then reheating it for about a day at some temperature below 300°C. The present work was undertaken to determine whether $Au_{82}Fe_{18}$ will become ferromagnetic as a result of an aging treatment at 200°C.

The spherical specimen previously measured in the quenched condition was requenched from 800°C into cold water and then aged for 22 h at 200°C. The susceptibility was measured with a vibrating sample magnetometer in a field of 10 Oe as a function of temperature from 4.2 to 235 K. After zero-field cooling to 4.2 K the measuring field was applied continuously during the entire sequence of measurements, while the temperature was allowed to rise slowly and continuously. The measurements were timed, and the magnetization and temperature data were stored in digital form by a computer. Groups of ten readings each were made in 10-s intervals during the 20-min period when the specimen was in the temperature range 4.2—55 K. In the next period of 57 min, groups of five readings each were made in intervals of 30 s, while the temperature increased from 60 to 163 K. During the last period of 30 min, groups of 10 readings each were made in intervals of 30 s and the temperature rose from 167 to 235.6 K. Figure 1 gives representative data points in the entire temperature range. The external susceptibility is here again limited by the reciprocal demagnetization factor. The highest value of σ/H, measured at 165 K, is $1.38(10^{-2}$ emu/g Oe), while the reciprocal demagnetization factor, calculated from the lattice parameter at 300 K, as shown above, is $1.37(10^{-2}$ emu/g Oe). This agreement confirms the interpretation of the plateau of the external susceptibility of the aged spherical specimen of $Au_{82}Fe_{18}$ as a result of limitation by the reciprocal demagnetization factor.

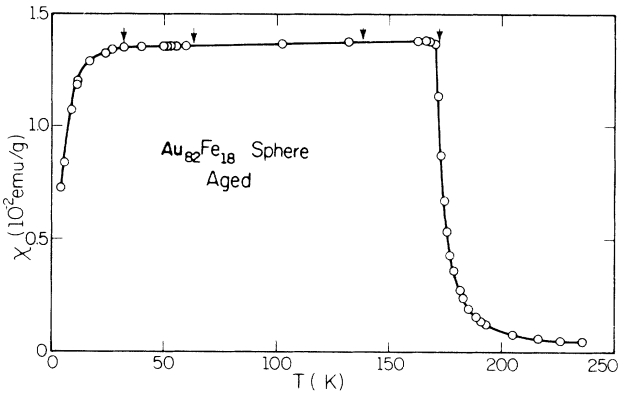

FIG. 1. Magnetization in a field of 10 Oe measured on the spherical alloy specimen as a function of temperature.

Figure 2 shows magnetization values measured again in a field of 10 Oe, but using a disk-shaped aged specimen of 3.5-mm diam and 0.05-mm thickness, held with its plane parallel to the field. Readings were taken in temperature intervals of 10 K. The temperature was changed automatically between measurements and held constant during the

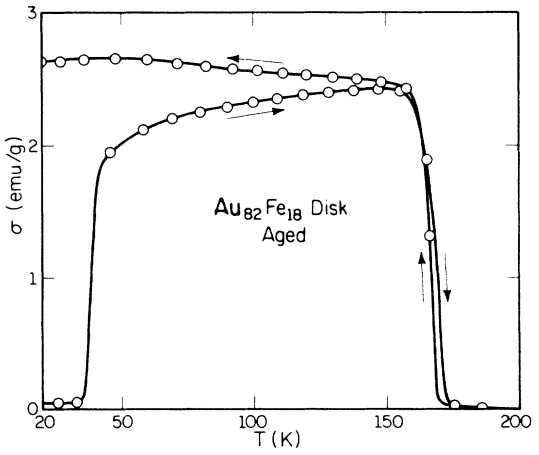

FIG. 2. Magnetization in a field of 10 Oe parallel to the plane of the disk-shaped specimen of 3.5-mm diam. and 50-μm thickness measured as a function of stepwise increasing and decreasing temperature, as shown by arrows. The field was turned off while the temperature was changing between measurements.

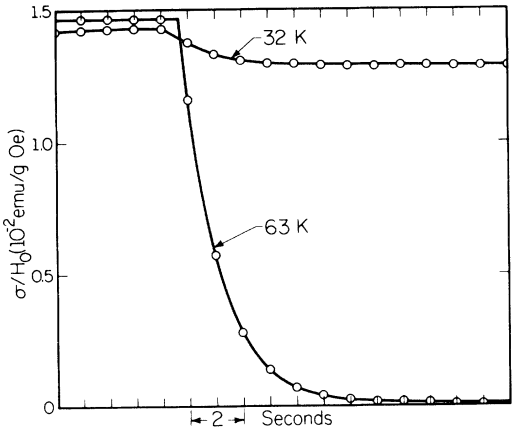

FIG. 3. Magnetization of the spherical specimen divided by the field initially applied as a function of time. Measurements made in 1-s intervals while the field was on, and also after it was disconnected. Magnetometer relaxation time was ~10 s. At 63 K remanence is very small. At 32 K it is about 90% of initial magnetization.

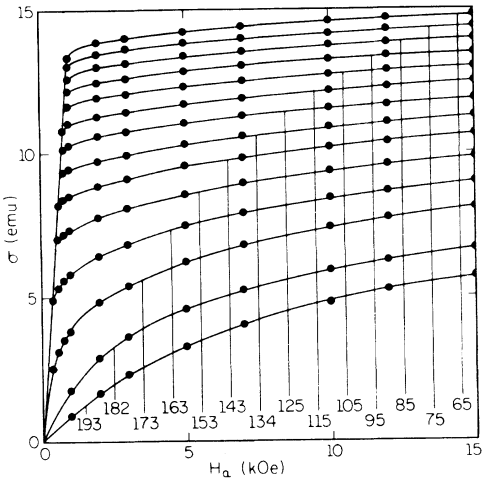

FIG. 4. Magnetization of 1.069-g spherical specimen as a function of applied field at temperatures indicated. In the temperature range used no remanence was measurable.

measurements under computer control. The time intervals between data points were approximately 3—4 min, during which the field was shut off. An increase of the maximum measured magnetization by a factor of about 19 is observed, as compared with the data in Fig. 1 for the spherical aged specimen of the same alloy. Because of the small thickness of the disk specimen, the demagnetization factor was so small that it had only a relatively slight influence on the external susceptibility, in spite of the high internal susceptibility of the aged specimen. This is shown both by the rounded form of the curves at the top and by the large difference between the ascending and descending temperature branches. The latter represents a large thermomagnetic history effect, characteristic of mictomagnets.[3] Interestingly, this effect is observed here even at temperatures where the remanence relaxation time is shorter than the time interval without field between data points. The difference between the two branches increases continuously with decreasing temperature, without any indication of a "phase change" or a "transition temperature." The largest external susceptibility values, $\chi_e=\sigma/H_a$, calculated from the magnetization measured in the vicinity of 50—60 K, is only about 0.26 emu/g Oe. Because of a small residual influence of the demagnetization factor noticeable here, the internal susceptibility χ_i may be larger than the external susceptibility by a factor of, at most, 3. Thus the maximum of χ_i may be estimated to be at most ~0.8 emu/g Oe. Although possibly as much as 30 times larger than the maximum of the internal susceptibility for the same alloy in the quenched condition, this value is still too low for a ferromagnet. Note that, in spite of this large difference in the maximum of the internal susceptibility, the maximum external susceptibility of the spherical specimen in the aged condition (calculated from the magnetization in Fig. 1) is the same as that in the quenched condition (Fig. 1, Ref. 3).

The relaxation of the remanence in the aged spherical specimen was studied at four temperatures, indicated by vertical arrows in Fig. 1. The results of two of these are shown in Fig. 3. All magnetization values are here divided by H_0, the field initially applied; at 63 K a field of 20 Oe was used and at 32 K a field of 10 Oe was used. After the field is shut off, at 63 K the remanence disappears almost completely in the first 10 s. After that time there is a period of very slow further decrease of the remaining small remanence. At 32 K the situation is similar, except for the much larger value of the very slowly decreasing remanence, remaining after 10 s. For the two higher temperatures studied, the curves obtained were almost identical with that for 63 K. The only difference was the complete disappearance of the remanence after 10 s at higher temperatures, as far as it could be determined at the sensitivity of detection available in this experiment. The temperature independence of the initial period of about 10 s after the field is cut off, during which the magnetization, measured initially with the field on, decreases to the level of the remaining, slowly decreasing remanence, if any, suggests that the duration of this initial period does not depend on the sample. Our observations indicated that it is, in fact, largely determined by the rate at which the output of the magnetometer is able to relax.

The remanence relaxation results show that even at temperatures well below 170 K [the temperature where the steeply rising external susceptibility of the spherical specimen fairly abruptly becomes temperature independent upon cooling (Fig. 1)], the aged $Au_{82}Fe_{18}$ alloy loses its magnetization as fast as can be measured after the magnet current is shut off. Only at 32 K, near the low-temperature end of the "flat top" of the curve, is there a large amount of remanence remaining 10 s after the field is removed. Apart from the thermomagnetic history effects shown in Fig. 2, above 63 K the alloy behaves essentially like a superparamagnet, but with an internal susceptibility so high that at temperatures below 170 K the external (measured) susceptibility of the spherical speci-

men is limited by the reciprocal demagnetization factor.

Figure 4 shows the measured magnetization isotherms as a function of the applied field H_a at the temperatures indicated. In order to eliminate transient effects connected with the magnetometer, after each step change in the field a waiting period of 15 s was allowed before making the measurements. At each temperature the field was at first increased and then decreased in several steps. The differences between magnetization values measured at the same field in the ascending and descending field sequences were less than 1% at temperatures up to 153 K and somewhat larger at higher temperatures. The data of the descending field sequences were actually used in Fig. 4. Both the temperatures and the fields were automatically controlled by computer. Each data point was the average of ten readings. The temperatures used (in approximately 10 K intervals) were above 63 K, where the remanence remaining 10 s after removing an initially applied field of 10 Oe was not measurable. The initial straight-line portions of all isotherms, except those for 173 K and for higher temperatures, were superimposed on each other. For the sake of clarity, many of the data points falling on the initial steeply rising straight line are not shown in Fig. 4. The superposition is a consequence of the fact that in the temperature range 65–163 K the susceptibility of the spherical specimen is limited by the reciprocal demagnetization factor not only at $H_a = 10$ Oe, as seen in Fig. 1, but even up to much higher applied fields, e.g., up to about 1 kOe at 65 K. As expected, the slope of the initial straight line in Fig. 4, expressed in terms of χ_e(emu/g Oe), agrees well with the calculated reciprocal demagnetization factor.

For the data points not on, or very close to, the initial straight line, the internal field H was calculated by correcting for demagnetization. Such $\sigma(H)$ data from pairs of adjacent isotherms were fitted with Eq. (1). The Brillouin function B was modified, as described in several previous publications, to take into account the interaction of the average magnetic cluster moment, $\mu = gS$, with its magnetic environment in the molecular-field approximation. For this purpose, a molecular field obtained by multiplying the dipolar portion of the magnetization $\sigma - \chi H$ with the Weiss molecular-field coefficient λ is added to the internal field H. Here χ is the temperature- and field-independent susceptibility. The parameter used to calculate the Brillouin function is then $\alpha = [H + \lambda(\sigma - \chi H)]/T$. Assuming that the orbital quantum number is $L = 0$, $g = 2$, and that S is not required to be an integer multiple of $\frac{1}{2}$, the Brillouin function can be expressed in terms of $\mu = gS$:

$$B = \frac{\mu+1}{\mu}\coth\left[(\mu+1)\frac{\mu_B}{k}\frac{H+\lambda(\sigma-\chi H)}{T}\right] - \frac{1}{\mu}\coth\left[\frac{\mu_B}{k}\frac{H+\lambda(\sigma-\chi H)}{T}\right]$$

and

$$\sigma = \chi H + \mu c B(\mu,\alpha) \,. \tag{1}$$

Here T, H, and σ are measured values for each data point, and the four parameters μ, c (the concentration of cluster moments), λ, and χ are to be adjusted by least-squares fitting to minimize the root-mean-square deviation (RMSD) of the calculated from the measured σ values. The root-mean-square *fractional* deviations attained in the present work ranged from 0.0024 to 0.0031, indicating a rather good fit of the data to Eq. (1) at all temperatures. The accuracy of the parameter values determined may be conservatively estimated to be about $\pm 5\%$.

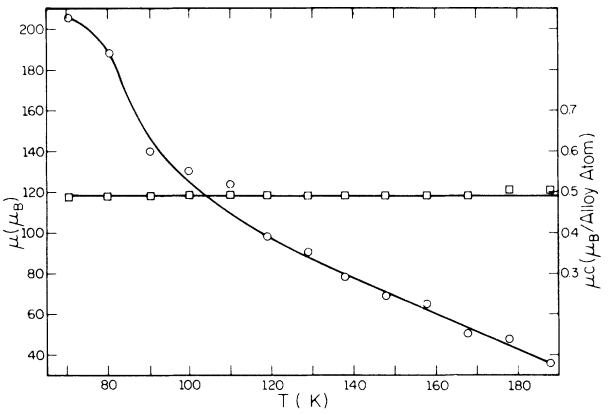

FIG. 5. Magnetic cluster moment μ and cluster concentration c obtained by least-squares fitting of Eq. (1) to data of pairs of adjacent isothermal magnetization curves shown in Fig. 4. The total cluster magnetization μc is quite constant in the entire temperature range.

As seen in Fig. 5, the "average" cluster moment increases from $36\mu_B$ at 188 K to $206\mu_B$ at 70 K. As for the quenched alloy,[2] the total moment of the clusters per alloy atom μc is quite constant in the entire temperature range at $\sim 0.5\mu_B$ per alloy atom. At 18 at.% Fe, this corresponds to a moment of 2.78 μ_B per Fe atom, an unusually high value. Borg[14] encountered an analogous problem with Mössbauer effect data. He proposed the explanation that in Au-Fe alloys with more than ~ 5 at.% Fe the Au atoms also acquire a moment, which increases with the Fe content. In order to account for the $0.58\mu_B$ per Fe atom excess moment over $2.2\mu_B$ in this way, the average moment per Au atom would have to be about $0.13\mu_B$ in $Au_{82}Fe_{18}$, either in the quenched or in the aged condition.

The λ values resulting from the fitting are positive, and they decrease with decreasing temperature from 12.6×10^3 Oe g/emu at 188 K to 2.18×10^3 Oe g/emu at 70 K. The χ values obtained are consistently small and negative, suggesting that the temperature- and field-independent paramagnetic contribution is negligible in the temperature range under study.

It should be noted that all the $\sigma(H)$ isotherms shown in Fig. 4 are of the same character, as emphasized by the fact that they all fit Eq. (1) to about the same degree of relative accuracy in the entire temperature range. None of these isotherms shows saturation up to 15 kOe. The high-field differential susceptibility (e.g., the slope of the curves between 12 and 15 kOe) decreases monotonically with decreasing temperature from 193 K, where the alloy is clearly paramagnetic (see Fig. 1) to 65 K, right through the

temperature range of steeply rising low-field susceptibility. The latter has been interpreted by various authors as indicating a phase transition to ferromagnetism. The data presented here do not support the idea of a phase change or of the occurrence of long-range ferromagnetism in the temperature range studied. As seen in Fig. 5, the data indicate rather clearly that *short-range* ferromagnetic spin order within the small iron-rich platelets is already fully developed in aged $Au_{82}Fe_{18}$ at 193 K. At this temperature the total moment of the magnetic clusters $c\mu$ already attains the same value ($\sim 0.5\mu_B$ per alloy atom) as at lower temperatures. What happens on further cooling to 70 K is that the average cluster moment *gradually* increases over the entire temperature range without any change in $c\mu$. There is no hint of a phase transition or Curie temperature in this temperature range. Rather, there is a gradual coalescence of the small magnetic clusters present at 193 K into increasingly larger magnetic clusters as the temperature decreases. Neither the steep increase at ~ 170 K, nor the "flat top" of the measured (external) low-field susceptibility versus temperature curve for the spherical specimen below 170 K (Fig. 1) has, in this case, anything to do with the onset of long-range ferromagnetism. As discussed previously, the nearly temperature-independent susceptibility is a result of the internal susceptibility becoming larger than the reciprocal demagnetization factor for the sphere when the average magnetic cluster moment increases to a sufficiently high value upon cooling. The magnetic behavior of this aged alloy, with its extensive short-range ferromagnetism, differs from that of a classical superparamagnet mainly by the occurrence of various irreversible and time-dependent effects (such as remanence and thermomagnetic history effects) and by internal susceptibility values unusually large for a classical superparamagnet.

The interpretation of the magnetic behavior of $Au_{82}Fe_{18}$ (either quenched or aged) in terms of short-range ferromagnetism is consistent with Murani's neutron-diffraction results,[6] which show that in $Au_{81}Fe_{19}$ the ferromagnetic spin correlations continue to increase on cooling to helium temperatures, as gradual freezing of the magnetic cluster moments continues. Although in his paper Murani is apparently considering long-range ferromagnetic spin correlations, it seems that the effects of increasing *short-range* ferromagnetic spin correlations on the integrated magnetic scattering intensity, which he measured, should be very similar.

The coalescence of small magnetic clusters into larger ones upon cooling, while the total cluster magnetization (μc) remains unchanged, may take place, for instance, according to the following model. If one assumes that the iron-rich platelets, discovered by Dartige, Bouchiat, and Monod[1] in quenched alloys, but according to H. Fraser[15] also present after aging for 22 h at 200 °C, comprise some Au atoms as well, it is easy to visualize that the latter will decrease the ferromagnetic exchange interaction between the Fe moments they separate. This could then result, depending on the number and the distribution of the Au atoms, in the splitting of a single platelet into two or more smaller magnetic clusters which are weakly coupled to each other. Such a complex cluster could then act at some higher temperature as two or more independent magnetic clusters with smaller moments, while at a lower temperature the interaction between some, or all, of the smaller clusters within a platelet may cause them to function as a single cluster with a larger moment. This model is consistent with the fact that the cluster moments derived from the magnetization data are smaller than those expected on the basis of the size of the iron-rich platelets, as discussed above.

It is significant that long-range ferromagnetism in $Au_{82}Fe_{18}$ is absent not only in the quenched state, but also after aging at 200 °C, a treatment that increases considerably the internal susceptibility, but preserves the arrangement of the Fe atoms in coherent platelets. It suggests the possibility that this type of spatial arrangement, the subdivision of the iron content into many small independent platelets, is responsible for the remarkable absence of long-range spin correlation in spite of an abundance of short-range ferromagnetism.

The absence of long-range ferromagnetism has the consequence that the earlier assumed "double phase transition" (paramagnet → ferromagnet → spin-glass) cannot take place in $Au_{82}Fe_{18}$. In view of the earlier results with $Au_{85}Fe_{15}$ (Ref. 3), it is also quite clear that the alloys with somewhat lower Fe contents will also not go through such a double phase transition upon cooling. One can be, therefore, reasonably certain that the so-called "reentrant behavior" is altogether absent from the fcc solid solutions in the Au-Fe alloy system.

In an earlier paper[16] it was noted that Fe-Al alloys of the Fe_3Al ordered structure in the composition range between 27 and 30 at.% Al have a nearly temperature-independent susceptibility within a certain temperature range, and that at lower temperatures they show thermomagnetic history effects characteristic of mictomagnets. I interpreted these observations at the time as indicative of a ferromagnet → micromagnet (spin-glass) transition upon cooling. In view of my findings for $Au_{82}Fe_{18}$, I now consider it quite probable that the nearly temperature-independent susceptibility in these Fe-Al alloys as well is a result of an essentially superparamagnetic internal susceptibility, large enough to limit the external susceptibility of a spherical specimen to the value of the reciprocal demagnetization factor.

ACKNOWLEDGMENTS

A large part of this work was done at the Materials Research Laboratory of the University of Illinois, with facilities support from Project No. DMR-80-20250. It is a pleasure to acknowledge the very valuable help in this phase from M. B. Salamon and in the earlier stages at the Colorado State University from C. E. Patton. I am very much indebted to L. Meichle and L. Ketelson for their continuing thoughtful help in introducing me to the intricacies of their computer-controlled magnetometer and to H. Fraser for communicating his unpublished electron diffraction results.

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