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Structural and magnetic properties of metastable fcc Cu-Fe alloys

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The structural properties of the metastable fcc Cu-Fe alloys obtained by high-energy ball milling of elemental powders have been studied by transmission electron microscopy. The same technique has been used to study the effect of thermal annealing on the alloyed samples. Experimental observations show that in some Cu-rich grains, even for short processing times, there is an Fe concentration up to about 30 at. % with an essentially flat concentration profile. In the same grains one observes also a relatively low density of dislocations, several small aggregates of point defects and small particles of oxide precipitates all having the same orientation with respect to the matrix. In a few cases it is also possible to observe a lamellar structure with a periodicity of a few nm which has been interpreted as arising from spinodal decomposition. The same origin has been attributed to structures modulated on a similar length scale, observed quite frequently in the annealed samples. All these observations suggest that during plastic deformation there is a transient supersaturation of point defects which increases considerably the atomic mobility and the kinetics of the solid-state reactions in the deformed grains, and that the driving force for alloying arises from the mechanical energy supplied to the sample by the ball impact. It is also shown that the observation of (partial) spinodal decomposition, prior to and after annealing, is compatible with the magnetic properties of these samples if one accepts that Fe atoms in fcc Fe-Cu alloys are present in two electronic states, as observed in other fcc iron-late transition metal alloys. [S0163-1829(96)00625-X]

INTRODUCTION

The experimental evidence[1] that high-energy ball milling is able to induce interdiffusion and alloying in a system like Fe-Cu, characterized by its highly positive enthalpy of mixing, has stimulated a lively debate about the physical origin of the thermodynamic driving force enabling the alloy formation. At the same time, the thermal decomposition of the metastable solid solution has been the subject of several investigations dealing mainly with anomalies observed in their magnetic properties.[2–4]

Experimental data, collected by x-ray diffraction (XRD),[5] differential scanning calorimetry (DSC),[5] magnetic measurements,[2–4] and extended x-ray-absorption fine structure,[6] show that atomic level alloying occurs gradually during the ball milling process, suggesting that mixing proceeds through interdiffusion at the heterogeneous interfaces. The crystal structure of the alloy depends on the average Cu/Fe atomic ratio, being fcc (γ) on the Cu-rich side, bcc (α) on the other and biphasic for an Fe concentration ranging between 60 and 80 at. %.[5] The lattice parameter of the γ solid solution is slightly expanded relatively to pure Cu (Ref. 5) even though the atomic size of α-Fe is slightly smaller than Cu. The thermal data obtained by DSC confirm that the heat released by the alloys upon thermal decomposition is in reasonable agreement with the computed enthalpy of mixing, particularly in the α-phase.[7] Upon thermal treatment the metastable solid solution decomposes into the parent phases; however, XRD is not able to reveal the presence of γ-Fe, often observed as a metastable phase in the decomposition of dilute Cu(Fe) solid solutions, owing to the small difference of lattice parameter and to the peak broadening caused by the small crystal size and by the residual atomic strain induced by ball milling.

Mossbauer spectroscopy of equiatomic samples milled for long times[3,8] shows that the sextet of α-Fe is no longer visible but for a trace of it. In its place one observes a broadened sextet corresponding to a disordered magnetic alloy, attributed[9] to the fcc Cu(Fe) solid solution. Upon thermal treatment at increasing temperatures one observes[9] a progressive decrease of the broadened sextet and the growth of the spectrum of α-Fe. In addition, at all temperatures one observes also the presence of a nonmagnetically ordered phase identified as γ-Fe,[9] whose amount reaches a maximum after thermal treatment at 600 °C.

Magnetic measurements show that the low-temperature saturation magnetization M_s of an extensively milled equiatomic mixture is the same as that of a mechanical mixture of Cu and Fe, (Ref. 3) and that the Curie temperature T_C of milled samples depends on the Fe concentration decreasing to about 600 K at the equiatomic composition.[7] M_s measurements at 5 K, both in the as-milled state and after different heat treatments,[4] show a progressive decrease of M_s with increasing temperature of annealing even before any new phase is detected by XRD, thus pointing to a change in the

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electronic structure of the Fe atoms not associated with a change of crystal structure.

In order to find a physical justification for the alloying effect several hypotheses have been advanced. The experimental observation[5] that appreciable interdiffusion occurs only in the presence of considerable atomic strain and when the grain size is reduced to the order of 10–20 nm, has suggested that the interface energy and the strain field of the dislocations might provide the free energy necessary for alloying.[5]

A different approach has been proposed by Yavari and Desrè, who have advanced two different hypotheses. In a first paper[10] it was suggested that the presence of contaminants like oxygen might affect the relevant thermodynamic parameters like the enthalpy of mixing. As an alternative,[11] it has been suggested that enhanced solubility may derive from the Gibbs-Thompson effect at the sharp tips produced by the mechanical fragmentation of the parent phases. All these hypotheses are based mainly on indirect experimental evidence but, actually, high-resolution electron microscopy of Cu-Fe powders,[12] while showing the presence of a large density of defects like dislocations and grain boundaries, has not shown the presence of needlelike microstructures. The other explanations have been critically examined for the Cu-Co system where they have been shown not to be satisfactory.[13] In this case, in fact, the grain-boundary energy would allow mutual dissolution for a grain size of the order of 1–2 nm, which is one order of magnitude smaller than what is actually observed by XRD and by electron microscopy. It was also reported that no difference in the heat release measured by DSC could be detected using starting materials with different oxygen content. Finally, it was shown that the dislocation strain field could not provide a sufficient driving force owing to the fact that the atomic sizes of the two elements are quite close to each other, so that the elastic energy released by the segregation is negligible. These considerations point out that if the driving force is provided by a free energy increase of the parent phases by incorporation of structural defects, the alloying process should annihilate these defects or reduce substantially their energy. It was also suggested[13] that milling promotes the formation of coherent interfaces between Cu and fcc Co and that when their spacing is reduced to the order of 1–2 nm the free energy of the interfaces becomes high enough to provide the driving force for alloying. The multilayer spacing necessary to induce alloying is of the same order of the critical wavelength for spinodal decomposition;[14] it is then possible to describe this process, for which, as far as we know, no experimental evidence has been reported in the literature, as a sort of inverse spinodal decomposition, that is a process in which a homogeneous alloy is obtained from a coherent multilayered structure whose spacing is reduced beyond the critical wavelength by the plastic deformation.

From the above remarks it appears that several points related to the process of mechanical alloying and to the thermal decomposition of systems with positive heat of mixing, and to Cu-Fe in particular, are still obscure, so that further careful microstructural characterization may help to improve our understanding of these phenomena.

EXPERIMENTAL

Mechanically alloyed powders were obtained by milling in an air-cooled Spex 8000 mixer-mill the equiatomic mixture of commercially pure 99.8 at. % Cu and 99.5 at. % Fe powders from Koch Chemicals under an Ar atmosphere. The mill was equipped with 2 balls of 12.7 mm diameter and the ball-to-powder ratio was about 5 to 1.

Specimens for TEM observations have been prepared by embedding the powders in an Al matrix with the following procedure. A small amount of powder was placed between two Al foils having a thickness of 0.5 mm; this composite was plastically deformed in a hydraulic press up to cold welding of the Al foils. Since this method can, in principle, affect the powder microstructure, even if this effect is not expected to be relevant owing to the severe plastic deformation experienced by the powders during the milling process and to the considerable difference in the mechanical properties between the powder itself and the Al matrix, some specimens have also been prepared by embedding the powders in an epoxy resin. For both kinds of samples, discs of 3 mm diameter could be punched after suitable mechanical grinding. Further sample preparation has been performed by dimpling, while final thinning was performed by Xe ion-beam milling on a liquid-nitrogen-cooled stage. No evident microstructural difference was found between specimens prepared with either method. The samples have been observed with a Jeol 4000 FX transmission electron microscope operated at 400 kV and equipped with an EDS x-ray detector.

The observations have been performed on blends of equiatomic composition milled for 1 h, with the aim of studying the first stages of the interdiffusion process and on samples heat treated at 623 and 723 K after long term milling. Concerning the first set of samples it was observed that at this stage of the milling process the powders were quite inhomogeneous and with an average grain size in the μm range; among a majority of grains of the pure elements a few alloyed grains could be found with an fcc structure and an Fe concentration ranging from 10 to 30 at. %. Quite surprisingly, the Fe atoms appeared distributed uniformly without any appreciable enrichment at the heterogeneous interfaces. This fact is an indication that the atomic mobility during the ball milling process is strongly enhanced so that the interdiffusion process appears to be interface controlled. The microstructure of the sample showed the following features.

Oxide precipitates were observed in both phases, namely Cu_2O in the fcc grains and Fe_3O_4 in the bcc ones. The nature and the structure of the present oxides was ascertained by the analysis of several microdiffraction patterns. All the precipitates were iso-oriented with the matrix showing that a precipitation phenomenon from a supersaturated solid solution had occurred. This is shown in Fig. 1 where the diffraction pattern from a Cu-rich grain is reported. Besides the main spots of the γ-phase it is possible to observe the presence of reflections belonging to the Cu_2O structure showing a definite orientation relationship between the two phases. Since the oxygen is expected to derive mainly from surface oxidation of the starting powders, the presence of oxide precipitates in the interior of the grains shows that ball milling is able to modify, at least at some stage of the process, the phase equilibrium between these two elements. In fact, both

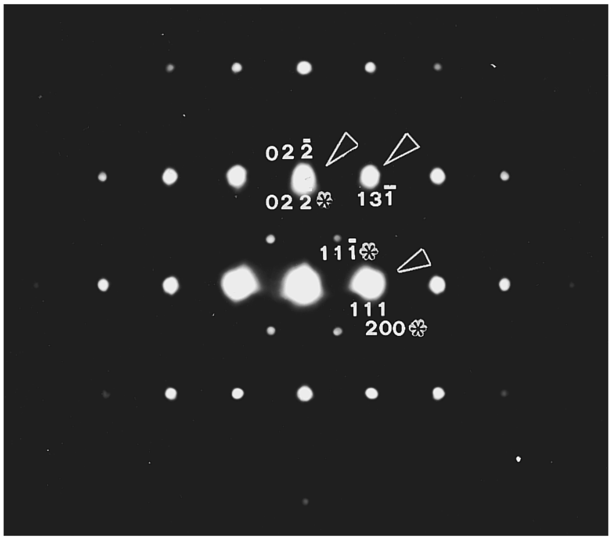

FIG. 1. Electron-diffraction pattern of a Cu-rich grain of a sample milled for 1 h showing the presence of Cu_2O particles precipitated semicoherently. The oxide spots are marked by an asterisk, while spots belonging to both structures are arrowed.

the initial and the final microstructures are coherent with the equilibrium phase diagram, while the change in the oxygen localization points to the temporary presence of a nonequilibrium Cu(O) solid solution.

The density of extended defects and, in particular, of dislocations is relatively low considering the severe plastic deformation imparted to the powders. This feature, shown in Fig. 2, suggests that some static or dynamic recovery of the defects induced by the plastic deformation has been operative during the process. This fact can probably be correlated with the high atomic mobility whose occurrence can be inferred from the uniform distribution of the solute atoms in the matrix.

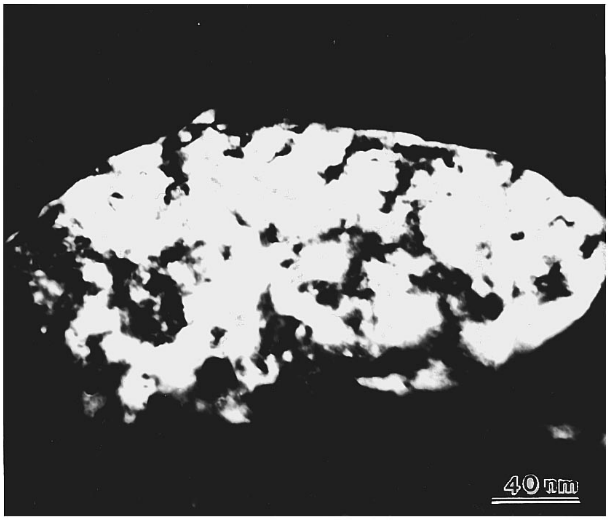

FIG. 2. Bright-field image of a Cu-rich grain of a sample milled for 1 h showing the presence of a small density of extended defects.

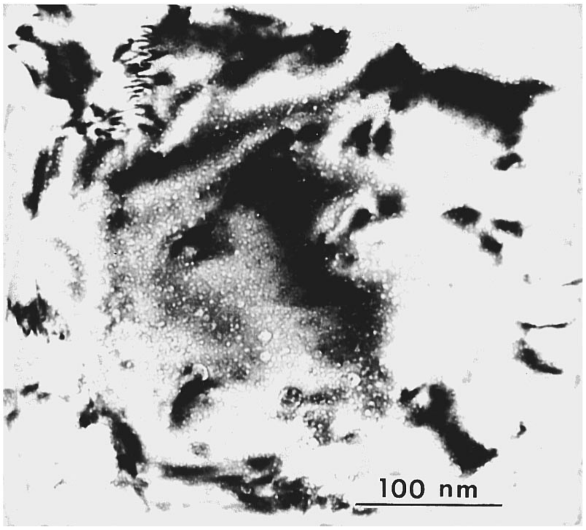

FIG. 3. Out of focus TEM image of a Cu-rich grain of a sample milled for 1 h showing the presence of small cavities.

Figure 3 shows an out of focus TEM picture of a Cu-rich grain where it is possible to observe a high density of intragranular voids, with a structure similar to that observed in irradiated materials. In fact the typical contrast of small cavities is observed both under focus and over focus.

In some Cu-rich grains with a Fe concentration around 30 at. %, one observes the modulated structure shown in Fig. 4(a). The contrast modulation has a periodicity of a few nm, typical of the critical wavelength in the spinodal decomposition of similar systems. Tilting experiments have shown that this structure is indeed lamellar since strong contrast in the TEM micrographs was observed only when the lamellas were parallel to the electron beam, so that a modulation was present also in the projected structure. This is shown in Fig. 4(b) where a dark-field image of the same area imaged in Fig. 4(a) is reported after tilting of about 10° around the [002] axis. The electron-diffraction pattern in the inset of Fig. 4(a) shows that while the grain is single-phase fcc with a [011] orientation, the contrast modulation is normal to a [111] direction. Considering that the crystal structure and the grain orientation do not change across the lamellar boundaries, this structure has been interpreted as arising from a spinodal decomposition of a supersaturated Cu(Fe) solid solution, where Cu-rich and fcc Fe-rich lamellas give rise to diffraction contrast in a TEM image owing to the difference in the average scattering factor and in the lattice parameter. In fact a mechanical refinement of the multilayers usually formed during ball milling is not expected to generate compositional modulations able to retain the same structure and orientation over the whole grain. These considerations are in agreement with the Mossbauer observations which show that some nonmagnetic Fe-rich γ-phase is always observed even in the as-milled state.

On the basis of the experimental data reported above as well as of the literature data mentioned so far, it is possible to evidence some features of the ball milling process as far as the crystal defects are concerned. It appears that there is a change, at least at some stage of the process, of the thermodynamic parameters so that the phase equilibrium is altered giving rise to unexpected reactions. On the other hand, decomposition reactions expected on the basis of the equilibrium phase stability are observed to occur. A possible explanation of this effect can be found in the impulsive nature of

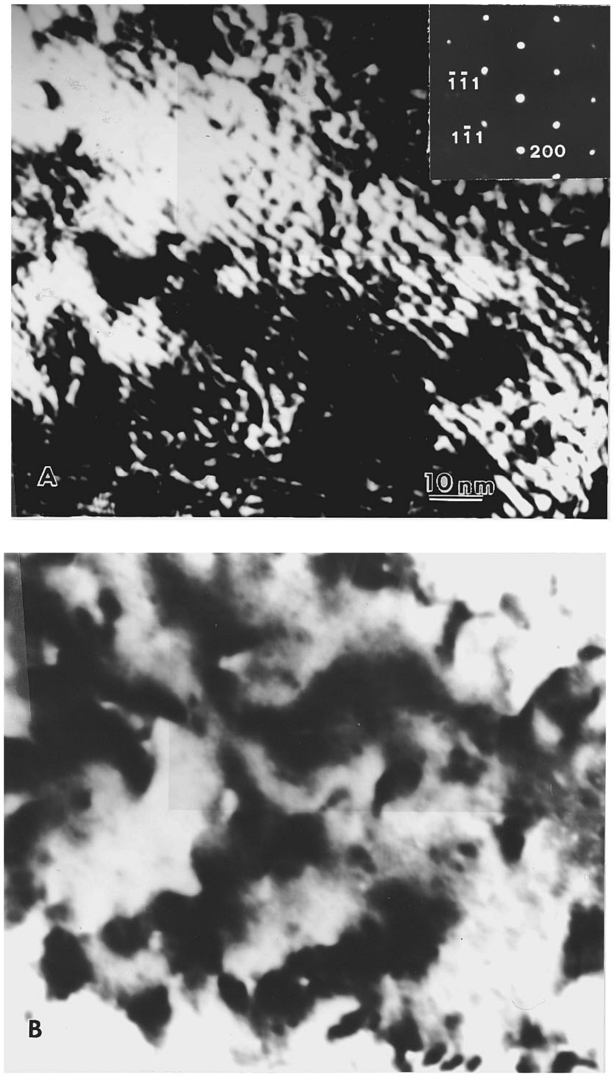

FIG. 4. (a) Bright-field image of a Cu-rich grain of a sample milled for 1 h showing contrast modulation roughly normal to a (111) direction. The grain is single phase as evidenced by the microdiffraction pattern shown in the inset. (b) Two beam dark-field image of the same grain tilted about 10° around the (002) direction. The magnification of both pictures is the same.

the deformation process inducing transient modifications in the sample structure. Probably several microstructural situations follow one another during a single impact between the colliding balls. The observed microstructure suggests that at an earlier stage both the atomic mobility and the phase stability are altered, while at a later stage only a high atomic mobility is observed.

Concerning the samples fully alloyed by ball milling and partially decomposed by thermal treatment, we have observed the presence of a large amount of modulated structures as reported earlier.[15] Electron microdiffraction experiments have shown that some of these grains are single-phase fcc with the same contrast features reported above, while in other cases two crystal structures, namely fcc Cu and bcc Fe, could be detected. The orientation relationships between the two phases are those typical of the martensitic transformation of fcc Fe.[15]

The microstructural observations in these samples allow us to formulate the hypothesis that the main mechanism of decomposition is spinodal, that is characterized by compositional fluctuations in the fcc matrix, and that the transformation of the Fe-rich layers to the stable bcc phase occurs martensitically when the local Fe concentration is sufficiently high.

DISCUSSION

The experimental results reported above show that none of the mechanisms proposed in order to explain the alloy formation in the Cu-Fe system during high-energy ball milling is compatible with the microstructure observed by TEM. Instead, our observations suggest that considerable atomic mobility is induced in the Cu grains by plastic deformation, at least at some stage of the process. It seems also probable that this high atomic mobility is due to a transient supersaturation of vacancies caused by the very high strain rate at which the deformation is carried out during high-energy ball milling.[16] This high vacancy concentration can thus explain the high atomic mobility necessary to produce the observed microstructure and can play a role also in the alloying mechanism.

It is also important to remark that all models proposed so far do not take into account the fact that the solid-state reaction occurs during the plastic deformation of the powders. Under these conditions the stress acting on the sample plays a role in the alloy formation since the work done by the external forces by displacing an atom contributes to the energy balance of the reaction. In this picture the stress field could be the source of the energy required by an Fe atom to enter substitutionally the Cu matrix. The mechanical energy ε_m supplied by the external stress to an atom moving a distance d along the stress direction is of the order of

$$\varepsilon_m = \sigma \pi r^2 d = \sigma V, \tag{1}$$

where σ is the stress intensity and r is the atomic radius. If d is chosen as the distance between two neighboring atoms or as the length of an atomic jump, V is of the order of the atomic volume. This energy contribution, in the presence of a suitable stress configuration at the Cu/Fe interface, can be used to force an Fe atom into the Cu matrix. According to Ref. 11, in order for Fe and Cu to form a continuous solid solution at 700 K, the free energy of the Fe atoms relative to the bulk relaxed state must be higher than the chemical potential of Fe at the spinodal point, calculated to be about 0.27 eV/atom.[11] In order for ε_m to be equal to this value, σ has to be of the order of 4×10^9 N/m^2. This value does not change substantially for all plausible values of d corresponding to values of V of the order of the atomic volume of Fe and compares very well with the maximum stress generated during ball milling, as calculated by Maurice and Courtney[16] in several cases. Considering that the above value of the chemical potential is relative to a substitutional solid solution, a vacancy in the Cu lattice is required in order for this mechanism to operate. A high vacancy supersaturation, induced by the external stress, is then required to enhance the kinetics controlling the atomic flux across the Cu/Fe interface. The agreement between the stress necessary to overcome the positive value of the chemical potential and that derived

TABLE I. The experimental (Ref. 4) saturation magnetization M_s in μ_B/Fe atom and the calculated γ_1, γ_2, and α percentages at two compositions, in the as-milled state (ams) and after thermal treatment at T_t.

		$Fe_{30}Cu_{70}$					$Fe_{50}Cu_{50}$		
T_t (K)	M_s	γ_1 (%)	γ_2 (%)	α (%)	T_t (K)	M_s	γ_1 (%)	γ_2 (%)	α (%)
ams	2.26	16	84	0	ams	2.18	19	81	0
623	2.06	24	76	0	623	2.05	24	76	0
723	2.00				723	1.73	31	44	25
823	1.39	37	0	63	823	1.29	41	0	59
923	1.79	19	0	81	923	1.71	22	0	78

from an analysis of the ball milling process supports the present hypothesis which accounts in a very simple way for the alloying of Fe and Cu during plastic deformation. While it is beyond our purpose to propose a detailed atomistic model for this process, we want to remark that the interface features can also play an important role acting as stress concentrators and that a contribution of the Gibbs-Thompson effect can be expected at the ledges created by the intersection of the slip planes with the interface itself.

Concerning the anomalous magnetic behavior of the metastable Fe-Cu alloys, the peculiar magnetic properties of iron atoms in the fcc lattice have been recognized for a long time.[17] In particular, it has been pointed out[18] that Fe atoms in magnetically ordered fcc alloys with late transition metals can exist in two electronic states γ_1 and γ_2, γ_1 being the low-energy state in the Fe-rich region. The transition from the γ_2 to the γ_1 state with increasing Fe content is particularly evident in the Fe-Ni alloys where it has been shown to lead to the ''invar'' effect.[18] The high volume ($a_0=0.365$ nm) γ_2 state is characterized by a high atomic magnetic moment, $\mu_a=2.8\mu_B$ and by ferromagnetic coupling; the low volume ($a_0=0.357$ nm) γ_1 state is characterized by a low atomic magnetic moment $\mu_a=0.7\mu_B$ and by antiferromagnetic coupling.[19] All these properties could be deduced from suitable extrapolations of existing alloy data and are in agreement with an experimental determination[20] of antiferromagnetism in small particles of γ-Fe precipitated coherently from a Cu(Fe) solid solution. Further experimental verification of the two-state hypothesis has been provided by a neutron-scattering experiment[21] on a ferromagnetic fcc $Fe_{70}Ni_{30}$ alloy, which has been interpreted as showing the coexistence of iron atoms with two magnetic moments, one with $\mu_a=2.7\mu_B$ ferromagnetically coupled to the bulk magnetization and the other with $\mu_a=0.4\mu_B$ antiferromagnetically coupled to the bulk magnetization. The existence of several local minima in the energy surface of γ-Fe as a function of magnetic moment and atomic volume has also been evidenced by modern total-energy spin-polarized band calculations.[22]

On the basis of these facts it is reasonable to suppose that, in agreement with the hypothesis of spinodal decomposition of mechanically alloyed Cu-Fe samples,[3,4] the magnetic anomalies observed in these alloys upon heating at different temperatures are due to the progressive transition of Fe atoms from the γ_2 to the γ_1 state, probably associated with temperature amplified concentration waves. Actually, the existence of a relevant fraction of magnetically disordered Fe atoms in an equiatomic Fe-Cu sample prepared by mechanically alloying and heated to 823 K, has been deduced from two Mossbauer spectra recorded at 8 K and at room temperature.[3]

In this context it is important to remark that the Néel temperature T_N of the γ-phase decreases with decreasing particle size starting from a ''bulk'' value of 67 K (Ref. 23) so that the hypothesis that the volume elements of the γ-phase are smaller than the minimum size detectable in an electron microscope offers a possible explanation of the Mossbauer observation relative to the existence of a nonmagnetically ordered state at 8 K. Moreover, considering that in a mean-field approximation T_N is proportional to the square of the spin magnetic moment, it is reasonable to suppose that in the present case the magnetic moment of the Fe atoms in the γ_1 state is small compared with its bulk value of $0.7\mu_B$. In the analysis of the magnetic anomalies, the exact value of the magnetic moment of the Fe atoms in the γ_1 state would then be of limited relevance since this value is in any case much smaller than that of the γ_2 state. As a consequence we adopt the hypothesis that the Fe atoms in the γ_1 state are nonmagnetic as reported in Ref. 3.

From the saturation magnetization measurements reported in Ref. 4 it is possible to deduce the percentages of the α, γ_1, and γ_2 state during thermal decomposition of fcc CuFe alloys with 30 and 50 at. % Fe. The low-temperature saturation magnetization in the as-milled state and after thermal treatment up to 623, 723, 823, and 923 K are reported in Table I in units of μ_B/Fe atom together with the α, γ_1, and γ_2 percentages. The γ_1 and γ_2 percentages have been deduced in the as-milled state and after thermal treatment at 623 K using the experimental observation[3] that in these samples no significant amount of α-Fe is present and assuming that the measured magnetization arises entirely from the atoms in the γ_2 state each having a ferromagnetically ordered magnetic moment of $2.7\mu_B$. The absence of a ferromagnetic γ_2 state in samples treated at 823 and 923 K has been observed by Mossbauer spectroscopy[3] so that in these samples one can directly deduce the amount of the nonmagnetic γ_1 state from the magnetization deficit relative to α-Fe. The data of Table I show that in the sample heated to 823 K approximately 40% of the Fe atoms are in the nonmagnetic state, compared to 20% of the resonant area in the Mossbauer spectrum and that a magnetization deficit of about 20% is present in the samples heated to 923 K. Concerning the samples treated at 723 K, they have been reported to contain both the α- and the γ-phase so that it is impossible to extract the γ_1 and γ_2 percentages without explicit knowledge of the amount of the α-phase present in the samples. An approximate value for this quantity can be deduced at the equiatomic composition from the XRD pattern recorded after heat treatment at 723 K and reported in Ref. 3. Recalling that at 923 K the α-phase comprises 78% of the Fe atoms of this sample, one has that at 723 K the α-phase is comprised between 20 and 30%, thus allowing us to deduce the percentages of γ_1 and γ_2 reported in Table I with no significant additional uncertainty. As evidenced in Table I the data relative to both compositions show a gradual transition from the γ_2 to the γ_1 state. At the same time there is also a transition to the α-phase, so that the percentage of the γ_1 state reaches a maximum after heat treatment at 823 K, as reflected in the value of the saturation magnetization. The percentages reported in Table I are similar for the two compositions showing that the transition from the γ_2 to the γ_1 state is essentially driven by the thermal treatment.

As mentioned above the transition is probably associated with spinodal decomposition of the originally ferromagnetic fcc solid solution and is consistent with the fact that pure γ-Fe has been found to be entirely in the γ_1 state.[20] Finally, from the present analysis of the magnetization data one deduces that a fraction of the Fe atoms are in the γ_1 state even in the as-milled samples, in agreement with the above reported experimental observation of (partial) spinodal decomposition.

CONCLUSIONS

The experimental results reported in this paper show that the alloying reaction between Fe and Cu cannot be explained on the basis of the microstructural features permanently present in the powders after milling. It should rather be attributed to transient modifications of the microstructure induced by the high amount of cold work imparted to the powders. These facts suggest that alloying occurs during plastic flow and is assisted on one hand by a transient supersaturation of point defects probably generated by the dislocation dynamics and, on the other hand, by the mechanical energy supplied by the external stress field. The scale on which concentration fluctuations are present in the samples has been only partially evidenced with direct imaging techniques. However, indirect support to the hypothesis of (partial) spinodal decomposition in the as-milled state and after thermal treatment comes from the analysis of the dependence of the saturation magnetization of the samples on the temperature of thermal treatment T_t. This analysis, based on the recognition that Fe atoms in the fcc structure can exist in two states, γ_1 and γ_2, having different magnetic properties and stable in different Cu concentration ranges, shows that as T_t is increased there is a first transition from the γ_2 to the γ_1 state and then a change of the Fe-rich phase from the fcc to the bcc structure. These changes are evidenced by the development of a minimum in the saturation magnetization and are probably caused by a progressive amplification of the concentration fluctuations up to a point where coherency between the Fe-rich and the Cu-rich fcc phases can no longer be maintained.

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