

Low-Spin  $\gamma$ -Fe-Ni ( $\gamma_{LS}$ ) in Fe-Ni Bearing Meteorites:  
Epitaxial Intergrowth of  $\gamma_{LS}$  and Tetrataenite as Possible Equilibrium  
State at  $\sim 20$ -40 at % Ni

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Abstract

We argue that the so called paramagnetic phase seen by Mössbauer spectroscopy in taenite lamella from octahedrites meteorites, ataxites meteorites, the metal particles of Fe-Ni-bearing chondrites meteorites, and synthetic particle irradiated Fe-Ni alloys is a low-spin  $\gamma$ -Fe-Ni phase ( $\gamma_{LS}$ ) related to the close packed low-spin phases seen in the pressure-temperature phase diagrams of both metallic Fe and synthetic Fe-Ni alloys and many other Fe-alloy systems. At a given composition, this  $\gamma_{LS}$  phase is quite distinct from the ordinary (high-spin)  $\gamma$ -phase ( $\gamma_{HS}$ ) in that it has a different electronic structure associated with very different magnetic properties (small-moment antiferromagnetism versus large-moment ferromagnetism) and a lower lattice parameter. It should be considered a new mineral for which we suggest the name antitaenite. We further propose that in the meteorites  $\gamma_{LS}$  always occurs in a fine epitaxial intergrowth with tetrataenite (atomically ordered FeNi). This resolves outstanding difficulties in meteoritic and particle irradiated Fe-Ni metallurgy.

**Key-words:** Meteorites; Tetrataenite; Low-spin  $\gamma$ -FeNi.

## 1. Metallic Iron and Synthetic Fe-Alloys

Experimental [1-4, and references therein] and theoretical [5-13] studies have established the existence of two close packed phases in the pressure-temperature (P-T) phase diagram of metallic iron: one (the  $\gamma$ -phase) has large magnetic moments, strong magnetic exchange interactions, and a relatively large lattice parameter, and another (the  $\epsilon$ -phase) has small or zero magnetic moments, weak or non-existent antiferromagnetic interactions, and a relatively small lattice parameter. Analogous phases have recently been shown to occur in the P-T phase diagrams of Fe-Ni alloys [14-16].

This shows that metallic Fe and Fe-Ni alloys can occur as two distinct phases having similar crystal structures (face centered cubic, fcc and hexagonal close packed, hcp) but with very different electronic structures: the ordinary high-spin ( $\gamma$ , here  $\gamma_{HS}$ ) phase and a high pressure low-spin (or possibly zero-moment)  $\epsilon$ -phase.

Analogous phases are seen in many interstitial and solid solution Fe-alloy series in that, under ambient conditions, a particular  $\gamma$ -phase (fcc) alloy is either  $\gamma_{HS}$  or  $\gamma_{LS}$  with moment per Fe-atom of  $\sim 2\mu_B$  or larger or  $\sim 0.5\mu_B$  or smaller, respectively. Non-magnetic stainless steels are examples of  $\gamma_{LS}$  Fe-alloys. The same phenomenon also occurs in amorphous Fe-alloys [17].

A  $\gamma_{LS}$  phase of metallic Fe (with a few percent Cu) can be stabilized in bulk amounts as coherent precipitates in a Cu matrix [18-20]. It has small magnetic moments that order antiferro- magnetically at  $T_N = 67$  K. The characteristic Mössbauer spectrum consists of a single (paramagnetic) line that experiences slight broadening corresponding to a small value of the hyperfine field at  $T < T_N$  [18]. The same  $\gamma_{LS}$ -phase metallic Fe can also be stabilized in epitaxially grown thin films [e.g., 21-25]. This is again consistent with the theoretical studies [5-13] that find that both the hcp and fcc structures of iron undergo high-spin to low-spin transitions at similar inter-atomic distances as the cell volume is decreased. The  $\gamma_{LS}$ -Fe that is stabilized by epitaxial interaction (or by alloying) is closely related to  $\epsilon$ -Fe of the P-T phase diagram but has a significantly different electronic structure from that of  $\gamma_{HS}$ -Fe, which is referred to as  $\gamma$ -Fe in the P-T phase diagram where it occurs only at high temperatures.

In addition, metastable precipitates of  $\gamma_{LS}$  phase Fe-Ni in a matrix of  $\gamma_{HS}$ -phase Fe-Ni (ordinary quenched fcc phase) of the same controlled composition have recently been synthetically produced near the Invar composition of  $\sim 35$  at. % Ni [26]. The latter precipitates caused magnetic domain wall pinning in the ferromagnetic matrix phase with a characteristic change in pinning strength at the  $T_N$  of the precipitates. This established the precipitate size as being at least the order of the domain wall width ( $\sim 2000$  Å). Since they were produced by cold working (i.e., by a diffusionless martensitic-like transition), they also have the same composition as the bulk matrix phase. Other studies using high field magnetometry [27-29] and neutron diffraction [30] also observed low temperature antiferromagnetism (in the range 34-45 at.% Ni) but interpreted this as intrinsic low temperature magnetism of the already ferromagnetic  $\gamma_{HS}$  matrix phase. When the  $T_N$  values from these studies and the domain wall pinning measurements [26] are plotted together versus bulk composition, they show a linear extrapolation from  $T_N = 0$  K at 48 at.% Ni to the known value of  $\sim 67$  K at 0 at.% Ni [26].

This is not an accident. The low field pinning measurements show that large precipitates that order magnetically at  $T_N$  are present and the extrapolation shows that these precipitates are the  $\gamma_{LS}$ -phase. Work on synthetic quenched Fe-Ni alloys therefore shows that metastable  $\gamma_{LS}$ -phase Fe-Ni is observed in the range  $\sim 30$ -50 at.% Ni. It would be difficult to

observe this phase at larger Ni compositions because of its absence of a Néel point at  $C > 48$  at.% Ni and it is not observed at  $C < 30$  at.% Ni because the quenched fcc alloys are unstable with respect to the fcc  $\rightarrow$  bcc (body centered cubic, or  $\alpha$ -phase) martensitic transformation at these compositions.

## 2. Meteoritic Fe-Ni

### 2.1 Review of key features

In the meteorite work, as with metallic Fe, synthetic Fe-alloys, precipitates, and thin films, Mössbauer spectroscopy has played a central role. In particular, Mössbauer measurements gave the first conclusive evidence [31-34] for the existence of a ferromagnetic atomically ordered FeNi (50 at.% Ni) phase that was later called tetrataenite [35].

Mössbauer spectra of: 1) taenite lamella from octahedrites [31-34, 36-39], 2) ataxites [40-48], and 3) the metal fractions of Fe-Ni-bearing chondrites [49-51], also show a single-line paramagnetic contribution at room temperature (RT) that is referred to as the "paramagnetic phase" or "paramagnetic  $\gamma$ -phase".

Recent detailed variable temperature measurements [47] of this phase in the Santa Catharina ataxite have shown line broadening that sets in at an ordering temperature of  $\sim 25$  K as temperature is lowered. At the lowest temperatures, the broadening is such that it must correspond to a very small saturation value of the hyperfine field. Under applied fields up to 80 kG [52-53] the hyperfine splitting remains small (with measured splitting essentially proportional to the applied field), showing that the small value is due to the electronic structure rather than dynamic effects such as superparamagnetism. Also, whenever the paramagnetic contribution is observed, it always coexists with a hyperfine sextet pattern that is unambiguously attributed to tetrataenite (atomically ordered or partially ordered FeNi phase, 50 at.% Ni) [31-34, 36-53].

The "paramagnetic" contribution is interpreted by the Mössbauer spectroscopists as being due to ordinary atomically disordered  $\gamma$ -phase (also called taenite and here called  $\gamma_{HS}$ ) whose Ni content is low enough for its magnetic ordering temperature to be much lower than RT. On the other hand, the most detailed analytic and crystallographic microstructural study of Reuter et al. [54] concludes that the paramagnetic phase seen by Mössbauer spectroscopy is most likely atomically ordered  $Fe_3Ni$  phase, in the clear taenite 2 (CT-2) structure of octahedrites. The same paramagnetic contribution seen in particle irradiated synthetic Fe-Ni samples (see below) is also interpreted as being ordered  $Fe_3Ni$  [55] but has been interpreted previously in terms of Fe-rich disordered  $\gamma_{HS}$ -phase [56]. Reuter et al. [54] have dropped their ordered  $Fe_3Ni$  interpretation in later work (personal communication with J.I. Goldstein).

These two interpretations (disordered  $\gamma_{HS}$ -phase and ordered  $Fe_3Ni$ ) are not easy to reconcile and are individually problematic. Consider the disordered  $\gamma$ -phase ( $\gamma_{HS}$ ) interpretation first.

## 2.2 Disordered $\gamma_{\text{HS}}$ -phase interpretation

Synthetic  $\gamma_{\text{HS}}$ -Fe-Ni alloys have never been produced that have such low magnetic ordering temperatures. The lowest observed Curie points ( $T_{\text{C}}$ ) are near RT. The lowest reported  $\gamma_{\text{HS}}$ -phase Fe-Ni  $T_{\text{C}}$  was measured in a two-phase ( $\alpha$ -phase and  $\gamma_{\text{HS}}$ ) alloy of composition 25 at.% Ni and found to be  $90 \pm 20$  K [57]. In addition,  $T_{\text{C}}$  decreases whereas  $T_{\text{N}}$  of  $\gamma_{\text{LS}}$ -Fe-Ni increases as Ni content decreases and has values that precisely match the observed meteoritic paramagnetic phase magnetic ordering temperature at the expected composition [47].

Most importantly, however,  $\gamma_{\text{HS}}$ -Fe-Ni alloys are ferromagnets with large atomic moments and large hyperfine field splittings such that far below  $T_{\text{C}}$  (at  $T/T_{\text{C}} \cong 0.2$ , say) even for low  $T_{\text{C}}$  values they must exhibit large hyperfine field splittings. The deviation from the Slater-Pauling curve of the saturation moment per atom in  $\gamma_{\text{HS}}$ -Fe-Ni as Ni content is decreased is due to antiferromagnetic alignment of some Fe moments rather than true local moment magnitude decrease and does not lead to significantly lowered hyperfine fields [58-59]. If the meteoritic paramagnetic phase were simply low Ni composition  $\gamma_{\text{HS}}$ -phase, then it should exhibit large saturation hyperfine field splittings. It would also have a relatively large lattice parameter that would make it observable as a distinct phase by X-ray diffraction [33-34, 36, 42, 46].

Another argument that the paramagnetic phase cannot simply be a low Ni content  $\gamma_{\text{HS}}$ -phase is that all such  $\gamma_{\text{HS}}$ -phase synthetic alloys are extremely susceptible to the martensitic fcc  $\rightarrow$  bcc transition. Quenched alloys in the range 25-30 at.% Ni that still contain  $\gamma_{\text{HS}}$ -phase rapidly lose it on lowering the temperature below RT. In contrast, the meteoritic paramagnetic phase is completely stable with respect to low temperatures down to  $T = 4$  K, which allows its magnetic ordering to be investigated. Explanations based on suppression of the martensitic transformation due to size or surface or epitaxial effects need to be invoked in order to explain the low temperature stability of the paramagnetic phase. Next, consider the  $\text{Fe}_3\text{Ni}$  interpretation of the meteoritic paramagnetic phase.

## 2.3 Ordered $\text{Fe}_3\text{Ni}$ interpretation

Whereas the existence of the atomically ordered FeNi and  $\text{FeNi}_3$  [60] phases is well established, no conclusive evidence has ever been obtained for the existence of an ordered  $\text{Fe}_3\text{Ni}$  phase in either synthetic alloys or meteoritic Fe-Ni.

If  $\text{Fe}_3\text{Ni}$  did exist, like its atomically ordered and disordered  $\gamma_{\text{HS}}$ -phase counterparts it should exhibit large hyperfine field splittings at low temperatures. Indeed, the paramagnetic contribution, with its small saturation hyperfine field value, is a very unusual and unique spectrum unlike that of any ordinary magnetic Fe-alloy. Only  $\epsilon$ -phase metallic Fe,  $\gamma_{\text{LS}}$ -phase Fe precipitates,  $\gamma_{\text{LS}}$ -phase Fe thin films, and "non-magnetic" low-spin Fe-alloys, have ever been observed to have similar Mössbauer signatures. The classical ferromagnetic, antiferromagnetic, and mixed exchange magnetism states of metallic Fe-alloys always lead to large saturation hyperfine fields ( $\sim 200$ -350 kOe).

## 2.4 Our proposed $\gamma_{LS}$ -phase interpretation

The above mentioned difficulties are resolved if we assign the meteoritic paramagnetic phase to  $\gamma_{LS}$  -phase Fe-Ni. Its magnetic ordering is the antiferromagnetic transition of the  $\gamma_{LS}$  -phase and  $T_N$  can be used to estimate its composition. Its low saturation hyperfine field value is a consequence of its low-spin electronic structure, it does not suffer from the martensitic instability which is a property of the  $\gamma_{HS}$  -phase, and its lower lattice parameter makes it unresolved (by diffraction methods) relative to the tetrataenite with which it coexists and which also has a lower lattice parameter compared to atomically disordered fcc  $\gamma_{HS}$  -phase. The  $\gamma_{LS}$  -phase (with estimated composition  $\sim 25$ -30 at. % Ni) has the same ambient conditions lattice parameter as tetrataenite (having various degrees of atomic order) in that the X-ray diffraction lines of the two phases have never been resolved in any meteorite sample. Both phases have smaller lattice parameters than their respective same-composition  $\gamma_{HS}$  -phase counterparts.

## 3. Synthetic Particle Irradiated Fe-Ni

When synthetic Fe-Ni alloys having 50 at.% Ni are irradiated with either electrons or neutrons, pure ordered FeNi phase (tetrataenite) is formed [61-62], however, when more Fe-rich alloys are irradiated a two-phase mixture is produced [55-56]. The Mössbauer spectra of the latter samples [55-56] show ordered FeNi ferromagnetic contributions (with various degrees of order depending on irradiation doses) and the same characteristic paramagnetic contribution ( $\gamma_{LS}$ ) that occurs in the meteorites, the thin films, the precipitates, and the high pressure work.

We therefore conclude that the paramagnetic contribution in Fe-rich irradiated Fe-Ni alloys is also  $\gamma_{LS}$  -phase Fe-Ni. This explains [55]: its lower lattice parameter (close to that of ordered FeNi), its lower saturation magnetization, its absence of a martensitic fcc  $\rightarrow$  bcc transition down to low temperatures, and its near-zero saturation hyperfine field splitting.

## 4. $\gamma_{LS}$ / Tetrataenite Epitaxial Intergrowth Equilibrium State

Given the above discussion, we propose that the meteoritic paramagnetic phase seen by Mössbauer spectroscopy is the  $\gamma_{LS}$  -phase. In analogy with the Fe precipitates and Fe thin films where the  $\gamma_{LS}$  -phase is stabilized at ambient pressure by epitaxial interaction with a matrix or substrate, respectively, and since the single-line paramagnetic phase seen by Mössbauer spectroscopy in both synthetic irradiated alloys and meteorites is never seen alone but always in coexistence with tetrataenite (having various degrees of atomic order, depending on the sample), we further propose that this  $\gamma_{LS}$  -phase always occurs in close microstructural association with tetrataenite.

Having studied the relevant literature [31-54, 63-65, and references therein], we conclude that the proposed  $\gamma_{LS}$ /tetrataenite intergrowth (IG) is a common state in slowly cooled meteorites, is the dominant state in both ataxites such as non-oxidized Santa Catharina

and in the CT-2 structures of octahedrites, and is present in the metal particles of chondrites. Such an IG has all the observed features of the meteoritic structures: 1) a single fcc lattice parameter because of the mutually stabilizing atomic scale matching between  $\gamma_{LS}$  and tetrataenite, 2) superlattice reflections corresponding to the three variants of the  $L1_0$  ordered FeNi structure, 3) Mössbauer spectra always showing the  $\gamma_{LS}$  -phase coexisting with tetrataenite, 4) average compositions always between those of tetrataenite and  $\gamma_{LS}$  -phase that would have the correct  $T_N$  value, and 5) outstanding stability of bulk amounts of  $\gamma_{LS}$  -phase due to the epitaxial nature of the IG.

A natural question arises: "Why has the IG never been seen by microscopic investigations?". We expect that the IG would be coarsest in the most slowly cooled meteorites (having structures of the right compositions). In fact, etched polished sections of the taenite particles in the Saint Séverin LL6 chondrite that is estimated to have cooled at a rate of 3 K/Ma [63] show outer taenite rims that have a unique fine structure consisting in a "totally unexpected...dense network of boundaries" [63]. This network resembles a two phase intergrowth and could not be easily interpreted by the discovering authors [63]. We propose that it is probably the first observation of the  $\gamma_{LS}$ / tetrataenite IG.

Our IG interpretation of common meteoritic structures suggests that, whereas at 50 at.% Ni the low temperature equilibrium state of Fe-Ni is probably tetrataenite which does occur as a pure single phase (e.g., in the CT-1 structures of octahedrites), at  $\sim 20$ -40 at.% Ni the low temperature equilibrium state is  $\gamma_{LS}$ / tetrataenite IG. This is noteworthy because the low temperature equilibrium phase diagram of Fe-Ni is problematic and a low-spin variety of the fcc structure has never been considered as a possible equilibrium phase [66, and references therein].

Also, we point out that, in connection with the applied pressure work on Fe and since  $\gamma_{LS}$ -Fe-Ni is a small lattice parameter state, we must expect it to be stabilized relative to  $\gamma_{LS}$  -Fe-Ni by applied pressures. This suggests that even the relatively low parent body pressures of the iron meteorites might have been large enough to influence the temperature, mechanism, and kinetics of  $\gamma_{LS}$ /tetrataenite IG formation. If  $\gamma_{LS}$  is a viable candidate as an equilibrium phase of Fe-Ni, then it becomes important to consider pressure in relating meteoritic observations to the temperature-composition ambient-pressure phase diagram of Fe-Ni.

Finally, note that mesosiderites (which cooled over 10X slower than the Saint Séverin chondrite) have never been studied by Mössbauer spectroscopy. These complicated breccias are believed to have resulted from impacts that shattered and mixed rocks on the surfaces of parent bodies. Nonetheless, when their metal fractions have mean compositions in the range  $\sim 20$ -40 at. % Ni, we might expect that the  $\gamma_{LS}$  would be present and that the  $\gamma_{LS}$  / tetrataenite IG would be coarse. Microscopic investigations, however, have not detected such an IG [ 67 and personal communication with J. I. Goldstein], similar to that seen in the Saint Séverin chondrite [63].

## 5. Conclusion

We offer a new perspective in which a low moment and small lattice parameter  $\gamma_{LS}$  -Fe-Ni phase is possible and is common in slowly cooled meteorites. Evidence for the existence of such a phase that is distinct from the high-spin ( $\gamma_{HS}$ ) variety is now abundant in

the areas of: the high pressure behaviour of metallic iron and Fe-Ni alloys (here it is hcp rather than fcc), coherent Fe precipitates in Cu and Cu-alloys, epitaxial thin films on Cu and other substrates, synthetic non-magnetic Fe-alloys, and synthetic Fe-Ni alloys. The  $\gamma_{LS}$ -phase is a unifying concept that resolves several outstanding problems in Fe-Ni-bearing meteorites and synthetic irradiated Fe-Ni alloys.

The key to realizing that  $\gamma_{LS}$  could occur as a distinct phase in Fe-Ni at ambient pressure and was not to be confused with the two- $\gamma$ -state ionic excitation model of Weiss that is often invoked to explain Invar behaviour was the observation that it could form precipitates large enough to pin magnetic domain walls in the matrix  $\gamma_{HS}$ -Fe-Ni phase [26]. This was the first demonstration that  $\gamma_{LS}$  and  $\gamma_{HS}$  Fe-Ni are true separate and distinct phases: the  $\gamma_{LS}$ -phase is antiferromagnetic at low temperatures whereas the  $\gamma_{HS}$ -phase is a collinear ferromagnet at  $C \geq 45$  at.% Ni and a non-collinear ferromagnet at  $C < 45$  at.% Ni that exhibits Invar behaviour at  $C \sim 35$  at.% Ni [26, 58, 68-69].

With this interpretation, a unique situation is seen to occur in both Fe-Ni-bearing meteorites and synthetic irradiated Fe-Ni alloys at  $\sim 20$ -40 at.% Ni: the  $\gamma_{LS}$ -phase seems to be in close epitaxial association with ordered FeNi (tetrataenite). The two phases have practically indistinguishable lattice parameters and form a very fine grained ( $< 0.1 \mu\text{m}$ ) intergrowth that we refer to as  $\gamma_{LS}$  /tetrataenite IG. This IG is indicative of the low temperature equilibrium state at these bulk compositions.

Finally, we believe our arguments are compelling enough that it is worth suggesting a name for this potential new mineral,  $\gamma_{LS}$ -phase Fe-Ni having  $\sim 25$ -30 at. % Ni and the same ambient condition lattice parameter as tetrataenite. Since it is a taenite and since its main characteristic that enables it to be identified (The Néel temperature is a known function of composition), we propose that it be called antitaenite. This also stresses its differences with ordinary (high-spin) taenite.

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