Formation of the Lamellar Structure in Group IA and IIID Iron Meteorites

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The Widmanstätten structure occurs in virtually all iron meteorites. The region between the Widmanstätten plates is the decomposed taenite, or plessite. Depending on the cooling cycle and composition of the meteorites, the plessite region decomposes into various morphologies. The lamellar plessite structure typically found in IA and IIID iron meteorites is of interest in this study. The lamellar morphology exists on two quite different scales, <1 µm and >3 µm as found in Group IA and IIID irons, respectively. This lamellar structure was characterized using analytical electron microscopy, light microscopy, and electron microprobe analysis. The α lamellae in Dayton (IIID) contained a compositional gradient from 6.1 ± 0.7 wt% Ni at the center of the α lamellae to 3.6 ± 0.5 wt% Ni at the α/γ interface. At this interface, the γ lamellae has a composition of 50 wt% Ni in α and 48 wt% Ni in γ. The orientation relationship at the α/γ interface in the lamellar regions of both Group IA and IIID has been determined by convergent beam electron diffraction and it conforms to the Kurdjumov-Sachs orientation relationship. This orientation is in contrast to the Nishiyama-Wassermann relationship that was found at the Widmanstätten pattern a/T interface. The phase transformations responsible for the lamellar structure in the IA and IIID chemical groups were examined. The IIID Dayton meteorite underwent a discontinuous transformation γα → α + γ between 400°-450°C. The phase transformation that produces the lamellar structure in the IA meteorites is not well established. Both an eutectoid reaction and a discontinuous reaction are feasible. Not until the role of carbon is understood or the existence of a high angle interface is observed will the identification of the phase transformation in IA irons become conclusive.

INTRODUCTION

Iron meteorites are composed primarily of Fe and Ni with small amounts of carbon and phosphorus and have cooled slowly in their parent asteroidal bodies. A Widmanstätten structure arises during cooling within an asteroidal body when kamacite (α) precipitates on the [111] planes of the prior taenite (γ). Upon cooling to low temperatures, the residual taenite regions between the kamacite decompose into various combinations of α and γ, usually termed plessite structures. The structure of the various plessite morphologies depends on the thermal history and composition of the meteorite. A unique lamellar plessite structure is found in carbon-containing Group I iron meteorites and one Group IIID iron (Dayton). The purpose of this research is to characterize the lamellar structure and to determine the phase transformations responsible for its formation.

Two forms of lamellar plessite (types A and B) exist in iron meteorites. Type A is a fine lamellar structure <1 µm wide, found in the high-Ni residual taenite of most Group IA meteorites. Representative meteorites from Group IA investigated in this study are: (A) Canyon Diablo, 7.1 wt% Ni, 0.26 wt% P; (B) Odessa, 7.35 wt% Ni, 0.25 wt% P; and (C) Toluca, 8.4 wt% Ni, 0.16 wt% P (Buchwald, 1975). Type B is a coarse lamellar structure >3 µm wide, found in the taenite matrix of the IIID Dayton meteorite. Several portions of this meteorite, containing 17.6 wt% Ni and 0.4 wt% P (Buchwald, 1975), were investigated. Groups IA and IIID contain very different nickel and phosphorus compositions. The carbon levels of Group IA are generally 0.2 to 2.0 wt% (Buchwald, 1975) while the carbon level in the IIID Dayton is 0.05 wt% (Moore et al., 1969).

EXPERIMENTAL PROCEDURE

Meteorite Sample Preparation

Meteorite samples for light microscopy and scanning electron microscopy were prepared using standard metallic polishing techniques followed by etching in 2% Nital seconds. Samples for electron microprobe analysis were also prepared by standard metallographic techniques, the samples were not etched. Scanning electron microscope (SEM) samples were coated with a thin layer of carbon to reduce charging effects. Meteorite samples to be examined using the transmission electron microscope (TEM) were first observed in the SEM in order to choose an area of interest, were then electro-discharge-machined into rods 3 mm diameter. These rods were then cut with a diamond discs of approximately 15 mil thickness. Final thin reduction of the discs was obtained by grinding to on 600 grit abrasive paper. The sample was then electroplated at +105V using a 2% perchloric/98% ethanol polishing at a temperature of -35°C.

Metallographic and Chemical Analysis

To understand the phase transformation responsible for the lamellar morphology, the structures of A and B were characterized using light, scanning, and transmission electron microscopy. Individual phase composition was determined by electron probe microanalysis (EPMA) X-ray analysis in the analytical electron microscope (AEM) the Cliff-Lorimer method (Cliff and Lorimer, 1974).
cal analysis of the lamellar structure was interpreted using Fe-Ni (Reuter et al., 1987a), Fe-Ni-P (Romig and Goldstein, 1978), and Fe-Ni-C (Romig and Goldstein, 1978) phase models. Convergent beam electron diffraction (CBED) was used to determine the orientation relationships of kamacite and taenite at the kamacite/taenite interfaces.

RESULTS

Structure of Dayton

The lamellar structure of Dayton varies extensively over an area of centimeters. As seen in Fig. 1, the lamellar morphology located around large phosphides (P, schreibersite) with Widmanstätten plates (W) running through it. The lamellar structure exhibits large variations in size and shape. One region surrounds the phosphide (schreibersite) and contains a few Widmanstätten plates, with length to width ratios. These large phosphides, which situate at high temperatures (~800°C), contain approximately 15.5 wt % P and 27 wt % Ni and are surrounded by a region of swathing kamacite. No cohenite inclusions were observed. A martensitic plessite structure is also found near the phosphide (M, Fig. 1). The second region is located approximately 1 cm away from the phosphide and contains a typical Widmanstätten structure.

Figure 2 shows the overlap of the two regions described at higher magnification. The Widmanstätten pattern (W) region contains plessite in a lamellar morphology (L). The lamellar structure in Dayton consists of parallel alternating kamacite and taenite phases. The taenite in the kamacite plates is either one-phase taenite or taenite with a "cloudy zone" sandwiched between two layers of taenite. The detailed structure of clear taenite and the cloudy zone as shown by electron microscopy in H is given by Reuter et al. (1987b). Figure 5 shows the variation of Ni across two taenite lamellae AEM. The compositional analyses of the kamacite and taenite at the kamacite/taenite interface are the same as those measured at the kamacite taenite boundary in the Widmanstätten pattern of the Dayton meteorite (Reuter et al., 1987b). Apparently, after nucleation, both phases, lamellar plessite and the Widmanstätten pattern, have similar microstructures while cooling to the same temperature. Buchwald (1975) argues on metallographic evidence that the two structures formed competitively at about the same temperature.

Microscopic orientation relationship between the kamacite and taenite 1 phases in the Widmanstätten structure. The lamellar structure was examined by using transmission electron diffraction (CBED) patterns obtained from γ regions of the Widmanstätten structure by traversing a sample under the beam show that the [110]_α and the [211]_α are parallel within very small errors (<1°) (Figs. 6a,b). Fig. 6 it is evident that the (002) diffraction vector in phase, g(002)_a, is parallel to g(022)_γ. From the CBED, the orientation relation is [110]_α//[211]_γ, (002)_a//[022]_γ, which is a Nishiyama-Wassermann orientation relationship. CBED patterns were also obtained for the α/γ interface in the lamellar structure. Figures 7a and 7b show CBED patterns from the [211]_α and the [121]_γ, indicating that the directions are parallel. From Fig. 7 it is clear that g(111)_α is parallel to g(101)_γ. Therefore, from CBED the orientation relationship is [211]_α //[121]_γ, (111)_α //(101)_γ, which is a Kurdjumov-Sachs (K-S) orientation relationship. Clearly, the orientation relationships of kamacite and clear taenite 1 phases in the Widmanstätten structure and lamellar plessite are different.
Fig. 2. Light micrograph exhibiting the Widmanstätten and lamellar morphology.

Canyon Diablo, Toluca, and Odessa—Group IA

The Group IA meteorites range from 6.5% to 8.5% wt % Ni, 0.2% to 2 wt % C, and 0.12 to 0.39 wt % P (Buchwald, 1975). Because of the lower bulk Ni values, the Widmanstätten α in Group I forms at higher temperatures and has thicker Widmanstätten plates than the higher Ni meteorite Dayton.

High Ni content in the residual taenite in Group I irons are caused by the impingement of Ni gradients due to Widmanstätten pattern growth.

The morphology of the plessite region varies extensively in the Group I meteorites. Figure 8 shows several types of lamellar structures that form in the residual taenite of the Group I meteorites. Figure 9 shows a plessite field that is partially cloudy zone and partially lamellar. The lamellar plessite in these micrographs appears to form at the clear taenite 1 boundary. The lamellar plessite in Group I irons occurs in areas of high Ni content. Electron probe microanalysis indicates the lamellar plessite forms in residual taenite regions contents between 25 and 38 wt %. For example, the structure intertwined with the cloudy zone has a content of 38 wt % Ni (Fig. 9). Other plessite are as acicular plessite fields had lower bulk Ni content wt % Ni).

The AEM was used to determine the composition < 1μm lamellar plessite. The lamellae contain a high phase of ~48% Ni and a low Ni BCC phase ~4 w The orientation relationship found at the kamacite interface of the lamellar structure is Kurdjumov-Sachs. the same relationship that was found at the kamacite taenite 1 interface in the lamellar structure of the meteorite, and also the same relationship observed the low and high Ni phases in the cloudy zone (Lbs 1977).

Fig. 3. Transmission electron micrograph of the one-phase taenite lamellae showing kamacite (K) and clear taenite 1 (CT1).

DISCUSSION

Dayton

Microstructure of Dayton. The phase transform that occur upon cooling of the Dayton meteorite understood by correlating the observed microstructure with the Fe-Ni-P phase diagram (Doan and Goldstein, 1970 and Goldstein, 1980).

The Dayton meteorite of bulk composition 17.6 % Ni and 0.4 wt % P was initially a single crystal of γ above and transformed to γ + Ph below 800°C. Figure 11 a schematic drawing of the P gradient developed as proceeded below 800°C. The length of the P concentration gradient is determined by the basic diffusion equation x = 2 \sqrt{D}t, where x is the distance of the P gras and t is time (~0.5 m.y.). At 70°C P concentration gradient is calculated as occurring ~

Fig. 4. Transmission electron micrograph of the three-phase lamellae showing kamacite (K), clear taenite (CT1), and cloudy zone (CZ).
cm. Another large phosphide would most likely nucleate within this P gradient, causing the profiles to overlap and depleting the matrix to the equilibrium phosphorous content between the schreibersite precipitates at 750°C (solid line, Fig. 10).

As the meteorite cooled from 750°C, the schreibersite expanded in size and depleted the matrix of P. The solubility of P and the diffusion distance in γ decreased. The P gradient forming from 750°C to 576°C across the γ from the schreibersite is represented in Fig. 10 with a series of hatched lines. Two γ regions appear to form: region A, which is relatively constant in phosphorus at ~0.2 to 0.3 wt %, and region B, which is close to the phosphide and is depleted in phosphorus by the continued growth of this schreibersite. The presence of such a P gradient in region B is also supported through microstructural interpretation (Fig. 1), which shows no α as well as large martensitic plessite fields. These fields form at low temperatures around the schreibersite. The martensite, α₂, forms when γ of low P content crosses the martensite start temperature, Mₛ, at ~250°C for the Dayton meteorite.

At 575°C, region A, containing 17.6 wt % Ni and ~0.2 wt % P, entered the α + γ + Ph phase field (Fig. 11a) while region B, 17.6 wt % Ni and < 0.2 wt % P (Fig. 11a), entered the α + γ phase field. The composition in both regions indicates that α is present. Small phosphides precipitated in region A at and below 575°C and helped nucleate Widmanstätten α (Narayan and Goldstein, 1984). At 575°C, the large schreibersite crystal would act as a nucleation site in region.
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affect only the precipitation sites and possibly the diffusion kinetics. Further X-ray analysis of the lamellar kamacite AEM shows a Ni composition of $6.1 \pm 0.7$ at the center of the lamella that decreases to $3.6 \pm 0.5$ wt% Ni at the kamacite interface. The decreasing Ni content from the center of the lamellar kamacite to the taenite interface indicates the lamellar structure precipitated at a temperature between $500^\circ$C and $400^\circ$C and then cooled to lower temperature. The depletion zone at the lamellar $\alpha/\gamma$ interface would no longer be present if precipitation occurred from martensite, $\alpha_2$, i.e., when low-$P_\gamma$ cools below the martensite temperature, $M_s$, of $250^\circ$C. In this case, the Ni content $\alpha$ would be well below 6.1 wt%. In addition, there is no evidence for the martensite, $\alpha_2$, structure to decompose upon cooling.

Two nucleation sites exist for the lamellar structure, small phosphides that formed throughout region A at the interface of the previously precipitated Widmanstätten plates and the $\gamma$ matrix in regions A and B. Figure 12a shows a schematic microstructure of regions A and B in a meteorite that developed at $575^\circ$C according to the precipitation process discussed above.

Between $575^\circ$C and $\sim400^\circ$C, region A remained in the $\alpha + \gamma + \text{Ph}$ field and region B remained in the $\alpha + \gamma$ field. At approximately $400^\circ$C the low-$P$ region B entered the $\alpha + \gamma + \text{Ph}$ region, and small phosphides precipitated. Parts of region B would undercool further until the martensite start temperature was crossed at $\sim250^\circ$C. These martensite areas are large in relation to the other martensite plessite fields in the meteorite (Fig. 1).

**Lamellar morphology in Dayton.** We have assumed that carbon has little or no effect on the Ni and P equilibrium compositions described by the Fe-Ni-P phase diagram for the Dayton meteorite. No carbides have been observed, optically or by electron microscopy, in Dayton in our study or by Buchwald (1975) either in the phosphides or in the Widmanstätten or lamellar structure. The Fe-Ni-C phase diagram (Römig and Goldstein, 1978) suggests C solubilities of $\sim0.25$ wt % in $\gamma$ to temperatures as low as $500^\circ$C. The Dayton iron contains 0.05 wt % C (Moore et al., 1969). Presumably carbon is present in solid solution in the taenite to temperatures below $500^\circ$C and perhaps below $400^\circ$C. We assume that C would have no significant effect on the precipitation of lamellar structure.
Light micrograph of the Toluca meteorite showing kamacite at taenite 1 (CT1), the cloudy zone (CZ), and the lamellar (L).

The analysis of the nucleation and growth mechanism interface between the lamellar phase and the Widmanstätten plate is difficult, however. Due to continued growth after nucleation of lamellar α, the probable α/γ interface as shown in Fig. 12b cannot be defined.

The various types of the discontinuous reactions are discussed by Williams and Butler (1981) and are generally defined by a discontinuous change in orientation of the product and a discontinuous change in solute concentration across the advancing interface. In Dayton, the discontinuous precipitation is described as the decomposition of a supersaturated single phase γ to a lamellar structure consisting of a precipitate (α) and solute depleted matrix (γ). The γ and γ are the same phase (taenite) and crystal structure (fcc), differing only in degree of solute (Ni) supersaturation. The reaction in Dayton is described as γ → α + γ, where γ is the matrix γ-taenite of Dayton containing ~17.6 wt % Ni and α + γ are the lamellar α and γ.

Canyon Diablo, Odessa, and Toluca

Composition of lamellar plessite in Group IA. In Group IA meteorites the Widmanstätten pattern began to form around 700°C. As the meteorite cooled, the α grew in width, while...
rejecting Ni into the $\gamma$. The rejected Ni built up at $\gamma$ interface due to the slow diffusion of Ni in taenite. A M-shaped profile developed. The Ni content in the core profile will be greater than the bulk Ni content in the meteorite because of impingement. The average Ni $c$ of the lamellar plessite in Group IA was measured at 0.05 % Ni by area scans in the EPMA. The solubility of Ni $c$ decreases to 0.04 wt % at 500°C, decreasing further with continued cooling (Romig and Goldstein, 1980). The $\alpha$ cohenite would precipitate at higher temperatures, an appreciable amount of carbon (~0.25 wt % C) (Romig and Goldstein, 1978) can remain in solution in $\gamma$ at 500°C.

**Fig. 12.** (a) Low magnification light micrograph of lamellar morphology and residual Widmanstätten plates in Region B of the Dayton meteorite. (b) Higher magnification of the intersection of the lamellae and the Widmanstätten plate.

This shows the lamellar structure and the cloudy zone be competing reactions. Actually, the lamellar structure probably formed at higher temperatures. The size of precipitates in the cloudy zone are $\sim$0.01 μm in contrast to the $\gamma$ lamellae, which are 0.1 $-$ 1μm in size. Re et al. (1987b) showed that the cloudy zone forms by a $\gamma$ reaction at temperatures below approximately 550°C. Diffusion of Ni is much faster at higher temperature. The lamellar structure probably precipitated at 400°C to 600°C and continued to grow until 300°C. At $\sim$300°C, the dissolution kinetics of Ni are so slow that the cloudy zone for the taenite lamellae through a spinodal decomposition.

As mentioned before, the retained taenite in Group I have an appreciable amount of C in solution at 500°C. As that the carbon precipitated as carbides above 400°C carbides could act as a nucleation site for the lamellar structure. Figure 12 shows the carbide mineral haxonite along $\alpha$ of the plessite field. This low-temperature carbide may form in nucleating the lamellar structure. Haxonite, however, seen in all lamellar fields. A three-dimensional examination of the lamellar plessite is needed to confirm this role of this as the nucleation site of the lamellar plessite.

Two types of nucleation mechanisms may control the structure formation; an eutectoid or a discontinuous reaction. An eutectoid-type precipitation involves the hetero-phase nucleation of the kamacite phase on a high angle $\alpha$ (such as haxonite). Upon formation the kamacite will Ni into the surrounding matrix; this will enhance the fo of the $\gamma$ phase or vice versa. The $\alpha$ and $\gamma$ lamellar will then advance into the $\gamma$ matrix. As indicated in the phase transformation that produced the lamellar morphology in Group IA occurred at approximately 520°C (Chuang et al., 1986) and Reuter et al. (1987a) proposed eutectoid transformation occurring at 500°C (Fig. 1 temperature of the reaction is in close agreement with work, but the predicted composition of the eutectoid is at 49.3 wt % Ni. This Ni composition is too high to directly applicable to the $\gamma$ phase in the lamellar s observed in Group IA. An appreciable amount of Ni present in the lamellar plessite area. The carbon may...
The lamellae consist of alternating plates of kamacite (α) and taenite (γ) with an interlamellar spacing > 3 μm. The α/γ interface of the lamellar structure conforms to the Kurdjumov-Sachs orientation relationship while the Widmanstätten pattern conforms to a Nishiyama-Wasserman orientation relationship.

The central region of the kamacite was a composition of 6.1 ± 0.7% Ni decreasing to 3.6 ± 0.5% Ni at the α/γ interface. At the α/γ interface, the Ni composition in the taenite is 50% Ni decreasing to 38% Ni in the center of the taenite.

The formation of the lamellar structure in Dayton, Group IID is dependent on the presence of large phosphide inclusions. Upon cooling of the meteorite, the schreibersite depleted the surrounding γ matrix of phosphorus. This phosphorus depletion caused the adjacent γ region to undergo a different phase transformation sequence than the rest of the meteorite. In regions away from the schreibersite, the typical sequence consists of the precipitation of small phosphides, in turn nucleating Widmanstätten α as the α + γ + Ph phase field was entered at 575°C. The P-depleted areas surrounding the schreibersite did not enter into the α + γ + Ph field, but undercooled into the α + γ phase field until 500-400°C. In this temperature range, and in the presence of carbon, the meteorite precipitated α in a lamellar morphology. The carbon is present in solid solution in γ, not in carbides. The phase transformation describing the precipitation of the lamellar structure in Dayton is discontinuous of the type γss → α + γ.

Dayton—Group IID

1. The lamellae consist of alternating plates of kamacite (α) and taenite (γ) with an interlamellar spacing > 3 μm.
2. The α/γ interface of the lamellar structure conforms to the Kurdjumov-Sachs orientation relationship while the Widmanstätten pattern conforms to a Nishiyama-Wasserman orientation relationship.
3. The central region of the kamacite was a composition of 6.1 ± 0.7% Ni decreasing to 3.6 ± 0.5% Ni at the α/γ interface. At the α/γ interface, the Ni composition in the taenite is 50% Ni decreasing to 38% Ni in the center of the taenite.
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Canyon Diablo, Toluca, and Odessa—Group IA

1. The lamellar morphology of the Group IA iron meteorites consists of alternating plates of kamacite (α) and taenite (γ) with an interlamellar spacing of < 1 μm.
2. The α/γ interface of the lamellar structure conforms to the Kurdjumov-Sachs orientation relationship.
3. The kamacite in the lamellar structure contains ~4 wt % Ni and the high Ni γ phase is ~48 wt % Ni.
4. The composition of the lamellar area is > 25 wt % Ni, < 0.04 wt % P, and < 0.25 wt % C. The Group IA lamellar transformation temperature is between 450° and 400°C.
5. It is unclear whether the lamellar structure in Group IA formed by a grain boundary discontinuous reaction or an eutectoid reaction. Until the identification of a high-angle interface is made, or the effect of C on the Fe-Ni phase diagram below 500°C is determined, the actual phase transformation cannot be conclusively identified.

Acknowledgments. The research was supported through NASA grant NAG 9-45. The authors wish to acknowledge Dr. Roy Clarke of the Smithsonian for his assistance in obtaining the meteorite samples.

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