Nedagolla, a remelted iron meteorite

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Abstract—The Nedagolla meteorite was recognized by Axon to be a rare example of an iron which has been preterrestrially reheated to the point of melting. The dendrite secondary arms are spaced 200 μm apart, implying that Nedagolla solidified and cooled at ~0.02°C/sec. The presence of (Fe, Cr)1-S inclusions precipitated during cooling in the interdendritic regions, and evidence of solute redistribution of Ni, Cr, Co, Si and P are consistent with this cooling rate. Such a rate indicates that Nedagolla cooled very near the surface of its parent body. Secondary microstructural features including the presence of isothermal taenite and minute phosphide precipitates, which have formed from the dissolution of primary phosphide material, indicate a later reheating to about 750°C for a period of several hours.

INTRODUCTION

Many meteorites have been either mechanically or thermally altered following their original formation. Jain and Lipschutz (1971) noted that nearly two-thirds of investigated irons contain effects indicating exposure to pressures ≥ 130 kb. Structurally, the degree of shock alteration ranges from the formation of Neumann bands to the remelting of metal. Axon (1962) has cited Nedagolla to be a unique iron meteorite which has been reheated to the point of melting. In this paper we have attempted to more accurately define the shock and thermal history of Nedagolla. This was accomplished by detailed metallographic and electron microprobe analyses and by the use of solidification and phase growth calculations.

BACKGROUND

The Nedagolla meteorite fell in 1870, and is preserved as a 5 kg mass (Hey, 1966). The bulk Ni content of Nedagolla as determined from chemical analysis is about 6.2 wt. % (Cobb, 1967, WAI and Wasson, 1970). The Ni content of kamacite determined by microprobe analysis is 6.0 wt. % and the P content is about 270 ppm (Reed, 1969). Axon (1962) observed a distinct pattern of slender dendrite skeletons in the heat altered zone of Nedagolla and concluded that the unaltered mass of Nedagolla must be dendritic. In the near-surface region, he identified a number of minute globules of silicate but no phosphides in the metal. In the unaltered region, the dendritic pattern was composed of clear areas of kamacite in a duplex ground mass. The clear areas were believed to be the Fe-rich cores of the dendrites produced during solidification. Axon concluded that Nedagolla was produced by complete melting and resolidification.

Besides being structurally unique, Nedagolla is chemically different from most iron meteorites. WAI and Wasson (1969, 1970) have found that although most irons contain less than 30 ppm Si in the metal; Nedagolla and Tucson are the exceptions, containing 1400 ppm and 8000 ppm Si, respectively. In addition, Nedagolla

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contains high Cr contents, 2600 ppm (WAI and WASSON, 1970), 2441 ppm (SMALES et al., 1967), and very low Ge contents, 0.005 ppm (WAI and WASSON, 1970). WAI and WASSON (1970) suggest that these meteorites formed under much stronger reducing conditions than the vast majority of the iron meteorites.

**EXPERIMENTAL**

Two etching reagents, a 1 per cent nitric solution and a 1 per cent aqueous sodium bisulfite solution, were used to optimize the information obtained from metallography. Fine structure was resolved with the scanning electron microscope. Both phase identification and quantitative analyses were obtained with the electron microprobe. Data was converted to chemical composition by the use of the ZAF correction program of GOLDSZTEIN and COMELLA (1969).

**RESULTS**

Two sections of Nedagolla were examined, USNM 2396 and 745. In USNM 2396 ablation deposits are preserved, and a visible heat altered zone extends inward from 2 to 4 mm from the outer edge of the section; in USNM 745 heat altered zones extend inward approximately 2 mm from the edges, with the greater fraction of the surface showing unaltered structure. The following data and results were primarily obtained from the unaltered regions of USNM 745.

*Dendritic structure*

The solidification of an alloy commonly occurs by the growth of primary crystals, dendrites, from the melt. The characteristic morphology of such crystals is one of branched, tree-like structures. Experimental observations (CHALMERS, 1964) indicate: (1) that dendritic growth takes place only when the melt is supercooled, (2) that growth directions are strictly crystallographic, and (3) that the spacing between the branches or arms is approximately regular and decreases with each successive order of branching. In the Fe–Ni system, dendritic growth takes place in the three mutually perpendicular cube directions.

In general, we found a uniform, equiaxed dendritic structure in sections of Nedagolla. Figure 1 shows the primary dendritic crystals, 1500 ± 200 µm wide, surrounded by a light matrix of interdendritic material. In contrast to AXON'S (1962) observations, no silicate inclusions were found in the sections we examined; V. F. Buchwald (private communication) reports that he has also been unable to find silicates. Also no free graphite or carbides were observed in our sections.

KOTLER et al. (1972) found that, although primary arm spacings can be influenced by any one of several growth variables, the spacings between secondary arms, which grow normal to the primary dendrite crystals, are essentially a function of the growth velocity. FLEMINGS et al. (1967) determined the effect of cooling rates on secondary dendrite arm spacings for Fe–Ni alloys. They found that as the cooling rate during solidification increases, the spacing between secondary arms decreases, and derived the relationship:

\[ d = 60 R^{-0.32}, \] (1)

where \( d \) is the secondary arm spacing in microns and \( R \) is the cooling rate in °C/sec. Equation (1) is based on dendrite arm spacings measured in a series of Fe–26 per cent Ni alloys experimentally cooled at rates ranging from \( 1 \times 10^{-3} \) to \( 1 \times 10^{4} \) °C/sec.
Fig. 1. The dendritic structure in the Nedagolla ataxite. Sections of the primary crystals appear as dark regions in a matrix of lighter interdendritic material. Etchant: 1 per cent NaHSO₃, field of view: 2·1 × 1·6 cm.

Fig. 2. A dendritic crystal with secondary arms in Nedagolla. The dashed line indicates the path along which microprobe analyses were performed. The markings near the edges of the photomicrograph are microhardness indentations used to register the area. Etchant: 1 per cent NaHSO₃, marker length: 200 µm.
Fig. 3. Concentration profiles of Ni, Cr, Si and P across secondary dendrite arms in Nedagolla. The profiles extend into the solute enriched regions between primary crystals.

Additional measurements by FLEMINGS et al. (1970) taken on Fe–10 per cent Ni and Fe–4 per cent P alloys indicate that solute content has relatively little effect on the relationship between cooling rate and secondary arm spacings. Measured arm spacings, in regions oriented to exhibit the branching of secondary arms from a primary crystal (Fig. 2) were 200 ± 50 μm. Using the average value, we estimate [equation (1)] that Nedagolla cooled, during solidification, at a rate of about 0.02°C/sec.

During solidification in a system where the solute element is known to lower the melting point of the alloy, the primary dendrites grow as relatively pure crystals rejecting solute into the surrounding liquid. After solidification, the microsegregation is such that a minimum amount of solute exists at the center of dendrite arms while the maximum concentrations occur between the arms. Using the electron microprobe, solute concentrations were measured along selected paths in Nedagolla. Figure 3 shows the result of concentration vs distance profiles for Ni, Cr, Si and P across the secondary arms of a primary crystal (Fig. 2). Each data point shown was obtained by recording X-ray intensities while the electron beam scanned a 50 × 40 μm area. In Fig. 3, note that (1) the valleys correspond to the centers of the dendrite arms, while the peaks represent the last regions to solidify; (2) the solute elements segregate more markedly in the region between primary crystals; and (3) the profiles for Ni, Cr and Si tend to correspond to each other while the P gradients are flattened. Since P diffuses in Fe more rapidly than either Ni, Cr or Si, P gradients have become more flattened during cooling to lower temperatures. The profile measurements for Co were similar to those of Ni, Cr and Si.

**Interdendritic sulfide and phosphide inclusions**

In Nedagolla, iron chromium sulfide inclusions were found in the regions between primary dendritic crystals. Although mainly globular, the sulfides also assumed morphologies which were influenced by the local solidification conditions. The compositions of nine sulfide inclusions were determined and are summarized in
Table 1. Analyses of (Fe, Cr)\textsubscript{1-x}S precipitates

<table>
<thead>
<tr>
<th>Element</th>
<th>Average conc. (wt. %)</th>
<th>Range of conc. (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>49.4 ± 0.5</td>
<td>40.9–58.1</td>
</tr>
<tr>
<td>S</td>
<td>40.5 ± 0.5</td>
<td>39.2–42.3</td>
</tr>
<tr>
<td>Cr</td>
<td>8.4 ± 0.2</td>
<td>4.9–11.6</td>
</tr>
<tr>
<td>Ni</td>
<td>0.4 ± 0.1</td>
<td>0.2–0.6</td>
</tr>
<tr>
<td>Co</td>
<td>0.1 ± 0.07</td>
<td>0.07–0.1</td>
</tr>
</tbody>
</table>

Table 1. The sulfides fall into the (Fe, Cr)\textsubscript{1-x}S phase field of the Fe–Cr–S diagram (EL GORESY and KULLERUD, 1967), with the value of ‘x’ varying from 0.10 to 0.21.

Figure 4a shows a scanning electron micrograph of a sulfide and the surrounding matrix, while Figs. 4b–d show S, Cr and P distribution in the same area. In Fig. 4a, a discontinuous phase approximately 1 μm in thickness is seen to rim the spheroidal sulfide inclusion. In addition, a number of globular micron size precipitates appear as white satellites extending as far as 12 μm from the inclusion. The metal matrix is kamacite and the lamellar structures are taenite. By correlating the scanning and X-ray micrographs, it is apparent that the matrix surrounding the sulfide precipitate is enriched in P and that both the discontinuous phase in contact with the inclusion and satellite particles in the matrix are phosphides. Several other sulfide regions were characterized by phosphide precipitation.

We propose that, following initial solidification and formation of the dendritic structure, phosphide formed around the sulfide inclusion. The satellite phosphides however resulted from a subsequent secondary reheating event which caused partial dissolution of the phosphide phase, and enrichment of the surrounding matrix in Ni and P. Upon cooling, a re-precipitation of the phosphides occurred resulting in the assemblage of phases seen in Fig. 4a.

Microstructure

The structure of the dendrite cores (Fig. 5a) consist of oriented lath-like taenite, 3–4 μm in width, in a structureless matrix of kamacite. The average composition of such regions is 5.7 ± 0.1 wt. % Ni and 0.05 ± 0.01 wt. % P. The morphology of taenite differs in the solute-rich interdendritic regions (Fig. 5b) which have average compositions of 6.75 ± 0.25 wt. % Ni and 0.11 ± 0.03 wt. % P. Taenite in these areas is irregular in shape, and measures less than 2 μm across. Small taenite, γ, particles are usually formed when a meteorite or Fe–Ni alloy composed of kamacite, α, or martensite, α\textsubscript{2}, is reheated into the α + γ phase field of the Fe–Ni diagram. This type of γ particle is conventionally known as isothermal taenite (BRENTNALL and AXON, 1962). The presence of taenite precipitates in both the dendritic and interdendritic regions of Nedagolla suggests that a secondary reheating event has occurred.

We have conducted heat-treating experiments on alloys of meteoritic composition in order to simulate the microstructures observed in Nedagolla. An alloy composed of Fe, 5.7 wt. % Ni, and 0.09 wt. % P, and a small section of kamacite from the Canyon Diablo meteorite composed of Fe, 5.9 wt. % Ni, and 0.23 wt. % P were used
The Ni and P concentration vs distance profiles across a typical taenite lath in a dendritic region of Nedagolla are plotted in Fig. 6. The Ni distribution assumes the shape of an 'M' profile with symmetrical peaks occurring at opposite edges of the lath. From such a plot, we can estimate both the peak temperature and the length of the secondary reheating event. By assuming that taenite nucleated from kamacite, grew at the peak reheating temperature, and cooled rapidly, the Ni concentration at the center of the laths should be an indicator of the temperature to which Nedagolla was reheated into (α + γ) phase field. The measured Ni concentration of ~ 6 wt. % corresponds to a peak reheating temperature of 750°C from the Fe–Ni phase diagram (Goldstein and Ogilvie, 1965a). Upon cooling from 750°C, the taenite laths become enriched in Ni at the α–γ interfaces, resulting in the M-shape profile observed.

The length of the reheating event can be calculated based on a model of diffusion controlled growth (Fig. 7a). For the taenite particles to grow, after nucleation from kamacite during the secondary reheating event, Ni must diffuse from kamacite into the precipitate. It should be noted that the rate controlling factor is the diffusion coefficient of Ni in α, since α and not the previous α2 structure is stable at the reheating temperature. The solution to such a growth problem is given by (Jost, 1952):

$$\xi = 2y \sqrt{D_\alpha t},$$  
(2)

where $\xi$ is the movement of the interface, $t$ is the time, and $D_\alpha$ is the diffusion coefficient of Ni in α.
coefficient of nickel in α. The constant γ is defined (Jost, 1952) from the boundary conditions of the problem by the relationship:

$$\gamma e^{\gamma^2}[1 - \text{erf} (\gamma)]\sqrt{\pi} = \frac{C'_a - C'}{C'' - C'}$$

where $C'_a$ is the Ni content in α before precipitation, and $C'$ and $C''$ are the respective Ni contents of kamacite and taenite at the interface. The diffusion coefficient $D_a$ is equal to $1.4 \exp(-58,700/RT)$ cm$^2$/sec, (Hirano et al., 1961) where $T$ is the temperature in °K. Figure 7a shows the concentration values used to determine $\gamma$ at 750°C. Solutions at 750°C show that 1–2 hr are required to grow taenite precipitates 3–4 μm in size. The Fe-Ni diagram indicates 800°C to be an upper limit for the peak reheating temperature, however, since kamacite containing 5.7 wt. % Ni ($C'_a$) will completely transform to taenite at this temperature. Solutions for 700°C based on equilibrium values of 9.8 wt. % Ni and 4.1 wt. % Ni for $C''$ and $C'$, show that 450–800 hr are required to grow taenite precipitates 3–4 μm in size.

As we have noted, both the nucleation and growth of isothermal taenite and the dissolution of phosphides associated with sulfide inclusions are the result of a secondary reheating event. In order to determine the accuracy of the temperature and the times we have suggested from considerations of the growth of isothermal taenite, further calculations were made based on a model of phosphide dissolution.
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(Fig. 7b). Of primary interest is the P concentration ($C_a$) as a function of distance ($x$) from the phosphide. Using equation (2) and the following equation,

$$C_x = \frac{C_a - C_a'}{1 - \text{erf}(\gamma)} + \frac{C_a' - C_a}{1 - \text{erf}(\gamma)} \left[ \text{erf} \left( \frac{x}{2\sqrt{D_t}} \right) \right],$$

(4)

from Jost (1952), $C_a$ as a function of $x$ and $t$ was calculated. The value of the ternary diffusion coefficient was calculated using $D_{FeP_a}^0 = 2.72 \exp (-52,200/RT)$, Heyward and Goldstein (1973), and the value of $C_a'(0.2$ per cent P) was that measured in the interdendritic regions of Nedagolla.

The calculations (Table 2) show that during a reheating event of 1–2 hr at 750°C,

Table 2. Calculated phosphide dissolution length ($\xi$) and P concentrations in kamacite ($C_a$) as a function of reheating time and distance $x$

<table>
<thead>
<tr>
<th>Reheating conditions</th>
<th>$\xi$ ($\mu$m)</th>
<th>$x = 6 \mu$m</th>
<th>$x = 12 \mu$m</th>
<th>$x = 18 \mu$m</th>
</tr>
</thead>
<tbody>
<tr>
<td>750°C, 1-2 hr</td>
<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>750°C, 2-1 hr</td>
<td>0.2</td>
<td>0.41</td>
<td>0.22</td>
<td>0.2</td>
</tr>
<tr>
<td>750°C, 5-0 hr</td>
<td>0.4</td>
<td>0.58</td>
<td>0.32</td>
<td>0.22</td>
</tr>
<tr>
<td>700°C, 450 hr</td>
<td>1.4</td>
<td>0.73</td>
<td>0.66</td>
<td>0.6</td>
</tr>
<tr>
<td>700°C, 800 hr</td>
<td>1.8</td>
<td>0.75</td>
<td>0.70</td>
<td>0.64</td>
</tr>
</tbody>
</table>

kamacite which originally contained 0.2 per cent P was enriched by as much as 0.2 per cent P at 6 $\mu$m from the dissolving phosphide and 0.02 per cent P at 12 $\mu$m. Since phosphide re-precipitation in Nedagolla is observed in kamacite to within ~12 $\mu$m from the original phosphide interfaces (Fig. 4a), a secondary reheating event at 750°C for 1–2 hr appears to be consistent with calculated results for the dissolution of phosphide and the growth of isothermal taenite. For longer heating times, 5 hr (Table 2), P enrichment will occur well beyond 12 $\mu$m. If secondary reheating occurred at 700°C for the same time periods as postulated for the isothermal taenite, P diffuses to considerable distances (>18 $\mu$m) from the original phosphide interface. Thus, reheating conditions at 700°C which allow for the formation of isothermal taenite seem improbable in the case of P dissolution.

**DISCUSSION**

Based on measurements of secondary dendrite arm spacings, we have shown that during solidification Nedagolla cooled at ~0.02°C/sec. Investigators (Wood, 1964; Goldstein and Ogilvie, 1965b) have estimated the sizes of the parent bodies of iron meteorites using cooling rate data. Models have been developed in which the parent body is composed of silicate material with a small metallic core. By assuming that cooling occurs by conduction through a homogeneous spherical body, the temperature at the center of the body after a time $t$ can be calculated from the equation (Carslaw and Jaeger, 1947):

$$T_c = T_i + 2(T_0 - T_i) \sum_{m=1}^{\infty} \frac{(-1)^{m+1}}{\pi^2} \exp \left( \frac{m^2\pi^2at}{\rho^2} \right),$$

(5)
where $T_0$ is the initial temperature of the body (°K), $T_s$ is the surface temperature (°K), $\alpha$ is the thermal diffusivity (cm$^2$/sec), and $r$ is the radius (cm). We used equation (5) to determine the amount of silicate, for example, needed to insulate Nedagolla during solidification and subsequent cooling. The values assumed for $T_0$ and $T_s$ were 1800 and 100°K, respectively, and the value for $\alpha$ was 0.01 cm$^2$/sec. To determine the radius $r$ of a body which would cool by conduction at a rate of 0.02°C/sec, equation (5) was differentiated with respect to time, and solved using several trial values of $r$. We found that 4—5 cm of silicate material would be all that were needed to insulate Nedagolla during solidification. This result could also be interpreted to mean that Nedagolla cooled very near the surface of a larger parent body. In all likelihood, collision processes were responsible for both the remelting and reheating of Nedagolla.

**Conclusions**

1. Nedagolla is composed of dendrites and interdendritic (Fe, Cr)$_{1-x}$S inclusions. Measurements of secondary dendrite arm spacings indicate that Nedagolla cooled at $\sim$ 0.02°C/sec.

2. Phosphide re-precipitation and the presence of isothermal taenite show that the event which caused remelting was followed by a later, less severe reheating to $\sim$ 750°C.

3. Calculations based on a cooling rate of $\sim$ 0.02°C/sec suggest Nedagolla cooled very near the surface of its parent 'body.'

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**References**


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