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AN ATOM PROBE STUDY OF PHASE DECOMPOSITION IN THE CAPE YORK METEORITE

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Abstract - An atom probe field-ion microscopy characterization of the Cape York iron-nickel meteorite has been performed. This investigation has revealed that an Fe-28 at. % Ni region in the taenite plate has phase separated to form an ultra-fine scale duplex microstructure. The results indicate the presence of composition modulations with a periodicity of approximately 10 nm and amplitude fluctuations from 50 at. % Ni to approximately 15 at. % Ni. In contrast, an Fe-20 at. % Nickel region revealed only minor fluctuations in composition.

1. INTRODUCTION

Iron-nickel meteorites are a unique material for studying phase decomposition in the iron-nickel system because of their extremely slow cooling rates of approximately 1°C per 10^6 years. This slow cooling rate enables low temperature transformations to proceed. The majority of iron-nickel meteorites have a characteristic Widmanstätten structure comprising of kamacite and plates of taenite.[1] Kamacite is a body centered cubic α-iron phase containing approximately 7% nickel in solid solution. Taenite generally refers to a face centered cubic austenite that contains more than 25% nickel. However, taenite plates are made up of several regions. Previous microprobe investigations have shown that the nickel profile across taenite plates has a characteristic M shape ranging from ~50% at the edge to less than 25% in the center of the plate. This profile arises as a result of the slow cooling process.[1] Several different regions have been identified within the taenite plate including a thin clear taenite (CT1) L1₀-ordered FeNi region at the edge, followed by a cloudy zone (CZ), a second clear taenite (CT2) region, and a plessite or a martensitic (α₉) region in the center.[2-4] The cloudy zone has been shown to be a mixture of globular L1₀-ordered FeNi in a honeycomb of either martensite, kamacite, or a disordered face centered cubic γ phase.[5] The cloudy zone varies in nickel content between approximately 45% near the CT1 region to approximately 28% adjacent to the CT2 region. The size of the globular L1₀-ordered regions decreases with nickel content. The CT2 region contains between approximately 28 and 24% nickel.

It has been suggested that the CT2 region is either an L₁₂-ordered Fe₃Ni phase or an ultra-fine scale mixture of L1₀-ordered Fe-Ni and martensite.[6] These two cases are difficult to distinguish with transmission electron microscopy since the extra reflections produced in electron diffraction patterns from the variants of an L1₀-ordered phase are equivalent to those produced in an L1₂-ordered crystal. In this paper, an atom probe field-ion microscope characterization of the Cape York meteorite is described with emphasis on resolving the identity of the CT2 region.

2. EXPERIMENTAL

The material used in this investigation was a sample taken from the Agpalilik mass of the Cape York Meteorite Shower. The 20,000 kg Agpalilik mass was found in 1963 by Buchwald.[2] The Cape York meteorite has been determined to be a medium octahedrite, Om, class and belongs to the chemical class IIIA.[2] The bulk composition of this iron-nickel meteorite was 7.40 at. % Ni, 0.47% Co, 0.27% P, 0.09% C, 2.2% S, 15 ppm Ga, 28 ppm Ge, and 2 ppm Ir.[1] The majority of the sulphur was consumed in troilite, FeS, and does not remain in solution. The cooling rate of this meteorite was determined to be approximately 3°C per 10^6 years.[7]
All atom probe analyses were performed in the ORNL energy-compensated atom probe field-ion microscope.[8] Field-ion micrographs were recorded with neon imaging gas and a specimen temperature of 60K. The atom probe results indicated that the elements conformed to the expected terrestrial isotopic abundances. This result is in agreement with other studies of the isotopic compositions where occasional differences have been revealed from those on Earth, but these differences were no larger than could be explained in terms of different cosmic radiation exposure or in different initial content of radioactive elements. Therefore, standard tables of natural abundances were used to determine the proportion of iron and nickel in the common isobar \((^{68}\text{Fe}^{2+}, ^{68}\text{Ni}^{2+})\) at a mass-to-charge ratio equal to 29. All compositions are quoted in atomic percent.

3. RESULTS AND DISCUSSION

The general microstructure of the Cape York meteorite is revealed in the low magnification optical micrograph shown in Fig. 1. The Cape York meteorite has a characteristic Widmanstätten microstructure consisting of an octahedral arrangement of kamacite and taenite plates known as octahedrite.[2] The bandwidth of this meteorite has been previously reported to be \(1.2 \pm 0.2 \text{ mm} \). The clear rim of an etched taenite plate is evident in the higher magnification optical micrograph shown in Fig. 2. In previous studies, similar \(~1\mu\text{m}-\text{thick~clear~rim~have~been~shown~to~be~the~tetrataenite~L}_{10}-\text{ordered~FeNi~phase.}\)[8,11]

Three field-ion micrographs of the kamacite and taenite containing either 20 or 28% nickel are shown in Figs. 3(a), 3(b) and 3(c) respectively. The field-ion micrograph of the kamacite revealed high quality images with the characteristic ring patterns of the planes being clearly visible. The taenite phase produced uniform field-ion images in which only the more prominent planes were resolved. The field-ion micrograph of the taenite plate containing 28% nickel showed some evidence of a fine-scale two phase microstructure in that the rings were not always continuous and a bright and dim contrast was discernible in some regions.

The variation in the nickel content across the taenite plates was determined in a scanning electron microscope (SEM). Nickel composition profiles from 20, 50 and 250\(\mu\text{m}\)-thick taenite plates are shown in Fig. 4. The three profiles exhibited the characteristic \(\text{M shaped profile with the minimum nickel levels in the center of the plates being approximately 21, 16, and 11\% nickel for the thin, medium, and thick plates, respectively. The maximum nickel content was found at the edge of the taenite plate. It is probable that the nickel content was actually higher than 35\% Ni measured in this SEM due to the excited volume of the electron beam collecting contributions from the adjacent low nickel kamacite. Using an electron microprobe in a study of 18 meteorites, Clark and Scott measured a mean composition of tetrataenite of 49.7\% Ni, 50\% Fe, 0.2\% Cu, 0.08\% Co, and <0.02\% P.}\)[11]

Atom probe analyses were performed on both kamacite and taenite regions. The results from four kamacite regions are summarized in Table 1. These atom probe results are in agreement with previous measurements.[1]

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Nickel</th>
<th>Cobalt</th>
<th>Phosphorus</th>
<th>Sulphur</th>
<th>Iron</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6.22\pm0.48</td>
<td>0.87\pm0.18</td>
<td>0.20\pm0.08</td>
<td>0</td>
<td>Balance.</td>
</tr>
<tr>
<td>2</td>
<td>6.32\pm0.34</td>
<td>0.46\pm0.09</td>
<td>0.34\pm0.08</td>
<td>0.02\pm0.02</td>
<td>Balance.</td>
</tr>
<tr>
<td>3</td>
<td>7.56\pm0.94</td>
<td>0.64\pm0.28</td>
<td>0.38\pm0.22</td>
<td>0</td>
<td>Balance.</td>
</tr>
<tr>
<td>4</td>
<td>6.94\pm0.16</td>
<td>0.62\pm0.04</td>
<td>0.10\pm0.02</td>
<td>0</td>
<td>Balance.</td>
</tr>
</tbody>
</table>

An atom probe composition profile through a region of taenite containing 20.0\pm0.57\% Ni, 0.39\pm0.09\% Co, 0.06\pm0.03\% C and 0.04\pm0.02\% Cr is shown in Fig. 5. This profile revealed only minor fluctuations in the nickel content. In contrast, a portion of an atom probe composition profile through a taenite region with a higher
overall content of 28.0±0.21% Ni revealed marked fluctuations in the nickel level, Fig. 6. This CT2 region was found to contain 0.56±0.04% Co, 0.27±0.02% C, 0.09±0.01% Cr, and 0.04±0.01% P. The nickel levels were found to fluctuate between a maximum of approximately 50% and a minimum of approximately 15%. These compositions would indicate a fine scale mixture of the equiatomic FeNi L1_0-ordered tetrataenite phase and a low nickel containing phase. This dual phase microstructure supports the observations from the field-ion micrographs. The periodicity of the composition modulation was determined to be approximately 10 nm.

Since no independent information was available, the identity (i.e. CZ or CT2) of the region analyzed has to be determined from the local composition or inferred from the observed microstructure. The two phase microstructure observed in the 28% analysis indicates that the volume analyzed was from the cloudy zone. However, the measured overall composition of 28% is within the experimental scatter for either the cloudy zone or a CT2 region. If this analysis was from the CT2 region, then the results support the two phase model rather than the single L1_2-ordered Fe_3Ni phase model. However, further analyses of taenite are required to verify which model is correct.

4. CONCLUSIONS

This atom probe field-ion microscopy investigation has revealed that an Fe-28% Ni phase in the taenite plate has phase separated to form an ultra-fine scale duplex microstructure. The results indicate the presence of composition modulations with a periodicity of approximately 10 nm and amplitude fluctuations from 50% Ni to approximately 15% Ni.

5. Acknowledgments

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6. REFERENCES

Fig. 1. Low magnification optical micrograph of the Cape York meteorite. The light regions are kamacite whereas the darker regions are taenite.

Fig. 2. Higher magnification optical micrograph showing darkly-etched taenite plates and kamacite in the Cape York meteorite. Note the light rim bordering the taenite plates.
Fig. 3. Field-ion micrographs of kamacite (a) and taenite containing 20 and 28 at. % nickel (b) and (c), respectively.

Fig. 4. Nickel composition scans across three taenite plates of different thicknesses showing the characteristic M-shaped profiles.
Fig. 5. Atom probe composition profile through a taenite region containing approximately 20 at. % nickel. This composition profile shows only minor fluctuations in nickel level.

Fig. 6. Atom probe composition profile through a taenite region containing approximately 28 at.% nickel. Phase separation into 50 at. % nickel and approximately 15 at. % nickel regions is evident.