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ABSTRACT

Ge distributions in 10 iron meteorites with bulk Ge contents of 8.7 to 2000 ppm have been measured by electron probe microanalysis. Ge is concentrated almost entirely in the metallic phases. It was redistributed in the temperature range at which the Widmanstatten pattern developed. Ge content shows a positive correlation with Ni content, reaching a maximum in taenite and a minimum in kamacite at the kamacite-taenite interface. The distribution coefficient of Ge between kamacite and taenite is relatively constant. The distribution of Ge is discussed in terms of its trace element behavior and its covariance with Ni.

THE DISTRIBUTION OF Ge IN THE METALLIC PHASES OF SOME IRON METEORITES

INTRODUCTION

One of the most debated subjects in meteoritics is the question of whether meteorites originated in one or several parent bodies. Goldberg et al. (1951) and Lovering, et al. (1957) have found evidence that iron meteorites can be grouped according to their Ga and Ge contents. Various authors have suggested that the Ga-Ge contents have a genetic significance and that meteorites with similar Ga-Ge abundances come from the same parent body. Recent measurements by Wasson (1967) have furthered refined the Ga-Ge groups and it now appears (Goldstein and Short, 1967) that several of these groups may have unique thermal histories.

Bulk Ga and Ge have been measured in many iron meteorites. However, little is known of the distribution of these elements within the metal phases kamacite and taenite or within the non-metallic inclusions schreibersite $(Fe\text{-}Ni)_3P$, troilite FeS, and cohenite $(Fe\text{-}Ni)_3C$. In this paper we report the results of a study on the Ge distribution in iron meteorites, and describe the geochemical behavior of this element during the formation of the Widmanstatten pattern.

METHOD

The distribution of Ge within a localized area (1 -5 sq. microns) of a meteorite can be measured non-destructively using an electron microprobe. The only limitation on such a measurement is the detectability limit of the instrument. The amount of a trace element C_x present in a sample is determined by the equation (Thieson, 1965),

$$C_x = \left[(\bar{N}_x - \bar{N}_B) / (\bar{N}_A - \bar{N}_B) \right] C_A \quad (1)$$

where $\bar{N}_x$, $\bar{N}_B$ and $\bar{N}_A$ are the mean counts determined for the unknown, its background, and the calibration standard respectively, and C_A is the composition of the calibration standard. From this relation, it is apparent that the detectability limit is governed by the minimum value of the difference $(\bar{N}_x - \bar{N}_B)$ which can be measured with statistical significance. Generally only about 200 ppm of most elements can be detected in a routine microprobe analysis.

This detectability limit of the probe is of limited value in the study of Ge distributions. Therefore, experimental procedures were developed, which lowered the detectability limit of our electron probe to 20 ppm at a 95% confidence level (Goldstein and Wood, 1966). This allowed measurements of meteorites in Ga-Ge groups I, II, and III (Lovering, et al., 1957).

To obtain a detectability limit of 20 ppm, a homogeneous calibration standard containing 1.1 wt % Ge (C_A), 5.1 wt % Ni and Fe was prepared. The Ge intensity $\overline{N}_A$ from this standard was measured before and after each determination of C_x. The background intensity for Ge, $\overline{N}_B$, was measured using homogeneous Fe–Ni standards (Goldstein, Hanneman, and Ogilvie, 1965) containing less than 10 ppm Ge. Measurements of the Ge concentration (C_x) in each area were made by repeated analyses of peak and background.

All the measurements were made with an ARL (Applied Research Laboratory) microprobe operating at 35kv and 0.2uA specimen current. The $Ge_{k\alpha}$ and $Ni_{k\alpha}$ radiation were measured simultaneously using LiF crystals. A total counting time of one hour was necessary for the detection of 20 ppm of Ge in the metallic phases.

Unweathered surfaces of all the meteorites studied were examined microscopically and a section was made perpendicular to one kamacite band system. This section was polished through 1/4 micron diamond and the oriented band system was identified. Ge was always measured in kamacite bands which were oriented perpendicular to the polished surface.

RESULTS

The Ge content of the minerals schreibersite (rhabdite), troilite and cohenite was measured in several meteorites. The inclusions were identified microscopically and compositionally with the probe. The inclusions and meteorites studied are listed in Table I. In these cases the background intensity, $\overline{N}_B$, for the inclusions was measured by analyzing the X-ray continuum intensity on both sides of the $Ge_{k\alpha}$ peak. The detectability limit in this case was 40 ppm. In all the inclusions analyzed, the Ge content was less than the detectability limit of 40 ppm. Therefore, almost all the Ge is concentrated in the metal phases of the meteorite.

Measurements of both the Ge and Ni distribution in the metal phases of 10 meteorites were made. One example of the Ge and Ni distribution in the kamacite, taenite, and plessite of the Butler meteorite, 2000 ppm (Goldstein, 1966) is given in Fig. 1. The data was taken by a point to point analysis and the relative precision was ±2.5% for the Ge. The Ni variation is typical of that found for the octahedrites. The Ni distribution in non-uniform in both kamacite and taenite.

In the taenite, the Ni content varies from a minimum of 16 wt. % Ni in plessite to almost 50 wt. % Ni in taenite near the kamacite-taenite border. In the kamacite, the Ni content is approximately uniform except near the kamacite-taenite border where the Ni is depleted.

The Ge varies directly with the Ni content of the metal phases. Therefore, during the cooling period in which the Widmanstatten pattern forms, there is a general movement of Ge from the growing kamacite phase to the parent taenite phase. In Butler, for example, the Ge content increases to over 4000 ppm in taenite at the kamacite-taenite interface.

The Ge and Ni contents of the other nine meteorites vary in the same way. The Ge and Ni distributions for those meteorites with more than 100 ppm Ge are shown in Fig. 2. Table II summarizes the data obtained for all the meteorites studied. The Ge contents measured in taenite at the kamacite-taenite interface ($C^{Ge}_{\gamma-Max}$) vary directly with the bulk Ge content of the meteorite. The ratio of $C^{Ge}_{\gamma-Max}$ to bulk Ge is about the same for all the meteorites studied, 2.0, with a standard deviation of 0.46. The Ge content in the center of the plessite, where the Ni content is a minimum, is also listed.

The Ge content of the kamacite varies directly with the Ni content (Figures 1 and 2) of the phase and also with the size of the phase. Detailed measurements of these variations have been made. To describe these measurements we must first define a few terms. The kamacite bands which have a bandwidth or plate thickness typical of the kamacite plates which make up the Widmanstatten pattern of the meteorite are defined as "average kamacite bands" hereafter referred to as (AKB).

The AKB are the first kamacite bands to nucleate as the meteorite cooled. They are usually the largest bands and used in classifying the octahedral structure of the meteorite. Kamacite bands smaller in size than the AKB nucleated in a temperature range below that in which the AKB formed. Normally these kamacite bands are about 10-50 microns in width and they will be called "low temperature kamacite" (LTK). To describe variations in Ge content from one kamacite plate to another, we will use the Ge content measured in the center of the phase.

The measured Ge contents of kamacite for 10 meteorites studied are given in Table III. The ratio of Ge in AKB to Ge (bulk) are also listed for those meteorites where the precision of the analysis was better than ±20% of the amount present. An average ratio of about 0.85 was obtained. In all cases, the Ge content of the average-sized kamacite bands was greater than that of the low temperature kamacite. Reproducible differences in Ge ($>$ 20 ppm) were measured for several AKB in one meteorite (Table IV). However, large differences were prominent only in Toluca.

Measurements of the Ge decrease in kamacite near the kamacite-taenite interface were difficult to make since severe Ge-Ni depletion only occurs about 10-20 microns from the interface, the amount of Ge depletion is relatively small and the diameter of the X-ray emission area at 35kv and 0.2μA from the electron probe is about 5 microns. Attempts were made to measure the depletion from three meteorites. In Canyon Diablo, the minimum measured Ge in kamacite was 150 ppm, a depletion of about 70 ppm from the AKB, in Odessa, 200 ppm, a depletion of about 60 ppm, and in Toluca, 110 ppm, a depletion of about 60 ppm.

It is interesting to note that the minimum measured Ge in kamacite for these meteorites is about the same as that of the low temperature kamacite.

DISCUSSION

Precision and Accuracy

Ge can be accurately measured in the ppm range since corrections to the measured data are unnecessary [Eq. (1)], and a well characterized Ge standard and Fe–Ni background standards were available. A reasonable average of Ge values determined for kamacite and taenite in our sample of 10 meteorites give approximately the same bulk Ge values measured by other investigators.

The precision of an analysis is given by the detectability limit (C_{DL}) for trace determination (Ziebold, 1965),

$$C_{DL} = \frac{C_A}{(\bar{N}_A - \bar{N}_B)} \left[st/\sqrt{n/2} \right] \tag{2}$$

were; n = number of measurements of sample and its background

t = student's distribution factor

s = estimate of total variance:

$$s^2 = \frac{\Sigma(N_x - \bar{N}_x)^2 + \Sigma(N_B - \bar{N}_B)^2}{2(n-1)} \tag{3}$$

For 95% confidence level, t can be specified from a "Students" Distribution Factor table (Fisher, 1950). The rest of the factors in Eqs. (2) and (3) can be measured. The measurements made in this study for a counting time of one hour indicate that C_{DL} = 20 ppm. The precision of an analysis ϵ, is therefore C_{DL} / C_x. The measured detectability limit includes the instrumental errors caused by instabilities in the high voltage, counting circuitry etc. of the electron probe and the error induced by the refocussing of the specimen in the light optics of the electron probe.

Comparison with Previous Measurements

Very few measurements of the Ge distribution in metallic meteorites have been made to date. In one study, Shima (1964) measured 122 ppm Ge in a troilite nodule of Odessa by a calorometric technique. Smales et al (1958) measured 32 and 19 ppm in two inclusions in the schreibersite and 30 ppm in the troilite of Sikhote-Alin by emission spectroscopy. The results of this study show that less than 40 ppm Ge is present in the minerals schreibersite, troilite or cohenite regardless of the bulk Ge.

The distribution of Ge in the metal phases of iron meteorites were measured spectrographically by Nichiporuk (1958). To determine the average Ge in kamacite and taenite, both kamacite and taenite were isolated chemically in dilute acid. His measurements indicated that Ge very strongly followed Ni. However, his Ge values are not correct. A comparison of the values measured for the same meteorites are given in Table IV.

Geochemical Behavior of Ge

The results of this study of Ge in iron meteorites are:

1. Ge is found in the metallic phases of the iron meteorites. The Ge content in schreibersite, troilite, and cohenite is less than 40 ppm.

2. Ge follows the Ni distribution in the kamacite and taenite phases. Bulk movement of Ge occurs at temperatures where the Widmanstatten pattern formed.

3. The Ge content in taenite at the kamacite-taenite interface varies with the bulk Ge of the meteorite. However, the ratio of $C^{Ge}_{\gamma-max}$ /Ge bulk is approximately constant at 2 for all the meteorites studied, which cover the range of Ge content from 2000 ppm to less than 50 ppm.

4. The Ge content of average-sized kamacite bands varies from one meteorite to another. The ratio of Ge in kamacite to bulk Ge is approximately 0.85 for all the meteorites studied having bulk Ge contents from 2000 ppm to 140 ppm.

5. The Ge content of low temperature kamacite is substantially lower than that of average-sized kamacite bands. The minimum Ge content in kamacite which occurs near the kamacite-taenite boundary is about the same as the Ge content of low temperature kamacite.

Measurements of Ge in chondrites indicate that at high temperatures (1500 °K) Ge behaves in a siderophilic manner. If this tendancy continues to temperatures where the Widmanstatten pattern forms, then Ge will remain in the metal at the same time that some of the non-metallic inclusions precipitate in the solid state.

The relations for Ge in iron meteorites as summarized by statements 2-5 are determined by the interaction of complicated thermodynamic and kinetic factors. Unfortunately, little is known about either. Thermodynamic data on third element impurities in the solid state is almost completely lacking and the Fe-Ni-Ge phase diagram has not been studied. In the binary system Fe-Ge, Ge acts as an α stabilizer, and is soluble in α to about 15 wt % (Hanson, 1958). In the binary system Ni-Ge, Ge acts as a γ stabilizer and is soluble in γ to about 15 wt % (Hanson, 1958). Therefore Ge, in the amount found in meteorites ($\leq$0.2 wt %) is soluble either in bcc kamacite or fcc taenite and can be considered as a trace element. As such, the Ge has little influence on the phase relations of kamacite/taenite. However, the relative amounts of Fe and Ni in kamacite and taenite greatly influence the distribution of Ge within the metallic phases. Therefore the Ge distribution is influenced by two main factors, bulk amount and the compositions of the metallic phases.

It is difficult to predict whether Ge follows the Ni or Fe gradients in the metallic phases since Fe and Ni are very similar geochemically, having similar valencies (Pauling, 1960) and atomic radii (Hume Rothery and Raynor, 1962). The fact that Ge prefers to be associated with Ni has been demonstrated by several experiments. In the first experiment Fe–Ni–Ge alloys with 5.1 wt.% Ni and either 1.1 wt.% or 0.5 wt.% Ge alloys were melted and solidified, the solidification process taking about one hour. When the alloys were analyzed with the electron probe, cored dendrites were found in which the Ge followed the Ni, increasing from the center of the dendrite to the edge of the dendrite. In a second experiment an inhomogeneous Fe–Ni–Ge alloy with 1.1 wt.% Ge and 5.1 wt.% Ni was annealed at 1300°C for varying amounts of time. The Ge followed the Ni, increasing as the Ni diffused from regions of high Ni concentration to regions of low Ni concentration. Evidently the presence of Ni lowers the activity of Ge, stimulating the Ge to follow the Ni content. The fact that Ge follows the Ni distribution in the metal phases of the iron meteorites, is consistent with experimental evidence.

At the kamacite–taenite interface, equilibrium is maintained to cooling temperatures of the order of 350°C during the formation of the Widmanstatten pattern (Wood, 1964, Goldstein and Ogilvie, 1965). At this interface the Ni content in taenite is a maximum ($C^{Ni}_{\gamma-Max}$) and the Ni content in kamacite is a minimum ($C^{Ni}_{\alpha-Min}$) (Reed, 1965; Short and Goldstein, 1967). The ratio ($C^{Ni}_{\gamma-Max}$ / $C^{Ni}_{\alpha-Min}$) measured with the electron probe varies between 6 and 10 although the actual ratio is probably the same for all the iron meteorites, about 10 ($C^{Ni}_{\gamma-Max} \simeq$ 50 wt %, $C^{Ni}_{\alpha-Min} \simeq$ 5 wt %). Therefore the partition ratio for Ge between kamacite and taenite, which is a function of the Ni content in the two phases, is constant with respect to bulk Ni and Ge. The actual amount of Ge in the AKB and the taenite at the two phase boundary is determined by the partition ratio for Ge and the bulk Ge content, C^{Ge}_{Bulk}.

It has been shown previously (Goldstein, 1965) that, even within one meteorite, the average Ni content of the kamacite bands varies with the width of the band. The variation of the Ni content of the band is a function of the temperature of nucleation of the band, the $\alpha/\alpha+\gamma$ boundary of the phase diagram and the width of the band. The Ni in the AKB which nucleate at high temperatures cannot equilibrate at low temperatures due to the large width of the band and the low diffusion rates. The Ni in the low temperature kamacite can equilibrate at low temperatures because the width of the band is small allowing more extensive diffusion. The variation of the average Ni content with kamacite band width determined by Goldstein (1965) is shown in Fig. 3. The AKB in each meteorite usually have a higher average Ni content than the LTK.

Since Ge follows the Ni gradient, it is entirely consistent that the Ge content is higher in the AKB than in the LTK. It has also been shown (Agrell et al, 1963) that a Ni decrease is observed in average kamacite bands near the kamacite-taenite boundary. This effect has been explained (Goldstein, 1965) as due to the decreasing solubility of Ni in kamacite below $\sim$ 450°C. The decrease in Ge near the kamacite-taenite interface of the AKB is consistent with the decrease in Ni content. The depletion effect occurs in the same temperature range as that for the growth of the LTK. Therefore it is also reasonable for the Ni and Ge contents near the α/γ boundary and in the low temperature kamacite to be about the same.

Since the distribution of Ge is dependent not only on thermodynamic factors but also on kinetic factors, it was thought that differing cooling rates might be an important factor in determining the Ge distribution. The cooling rates for the meteorites studied are listed in Table III. Although these meteorites differ in cooling rate by an order of magnitude, no correlation with Ge distribution can be seen. In another study it has been shown, however, (Goldstein and Short, 1967b) that bulk Ge and cooling rate are strongly correlated.

CONCLUSIONS

The Ge present in iron meteorites is concentrated in the metallic phases. Therefore measurements of the Ge compositions of meteorites for which large inclusions are avoided, will yield good representative bulk analyses.

Ge varies in the same way as the Ni reaching a maximum Ge content in taenite and a minimum Ge content in kamacite at the kamacite-taenite interface. It was redistributed during the period when the Widmanstatten pattern formed and the redistribution was dependent on a relatively constant partition coefficient for Ge between kamacite and taenite and the strong affinity of Ge for areas of high Ni content.

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Table I

INCLUSIONS ANALYZED

Mineral	Meteorite	Description
Schreibersite (Rhabdite)	Odessa	3 phosphides, ~ 45 wt. % Ni.
	Carbo	1 phosphide, ~ 45 wt. % Ni
	Grant	1 phosphide surrounding a troilite nodule ~ 25 wt. % Ni
	Canyon Diablo	1 rhabdite particle, ~ 41 wt. % Ni
	Butler	1 phosphide particle ~ 37 wt. % Ni 2 phosphide particles, ~ 50 wt. % Ni
Troilite	Grant	One inclusion
	Carbo	Two inclusions
Cohenite	Odessa	Four inclusions

Table II

Ge CONTENTS IN TAENITE

Meteorite	Structural Class	Ni (wt. %)	Ge (ppm)	$C^{Ge}_{\gamma\text{-Max}}$	$C^{Ge}_{\gamma\text{-Max}}$/Ge (bulk)	C^{Ge} for Plessite
Butler	Off-D	16.0[1]	2000[1]	4000±80	2.0	1550±80
Canyon Diablo	Og	7.24[2]	328[6]	430±40	1.4	200±40
Odessa	Og	7.35[2]	296[6]	460±40	1.5	210±40
Toluca	Om	8.31[2]	170[3]	380±20	2.2	170±20
Four Corners	Om	9.7[3]	141[3]	380±20	2.7	170±20
Hualapai	Of	12.3[4]	102[5]	180±20	1.8	30±20
Carbo	Om	9.98[5]	87[5]	155±20	1.8	110±20
Spearman	Om	8.4[2]	46[5]	—	—	25±20
Grant	Of	9.48[5]	38[5]	95±20	2.6	< 20
Carlton	Off	12.68[2]	8.7[5]	25±20	—	> 20

[1] Wasson (1966a)
[2] Goldberg et al. (1951)
[3] Lovering et al. (1957)
[4] Goldstein and Short (1967)
[5] Wasson (1966b)
[6] Wasson (1966c)

Table III

Ge DISTRIBUTION IN KAMACITE

Meteorite	Ge (bulk)	Ge in Kamacite Average Kamacite/Low Temperature		Ratio Ge in AKB/Bulk	Cooling Rate (Goldstein and Short, 1967a)
		Bands	Kamacite		
Butler (Off-D)	2000	1700, 1890, 1760 1735, 1600±80	1520, 1520, 1490 1480, 1460±80	0.85	0.5°/10^6 Years
Canyon Diablo (Og)	328	215, 225±40	105, 105±40	0.7	2.5°/
Odessa (Og)	296	250, 255, 250, 305±40	185, 195±40	0.90	—
Toluca (Om)	170	140, 170, 200±20	110±20	1.0	1.6°/
Four Corners (Om)	141	125, 130±20	110±20	0.9	1.9°/
Hualapai (Of)	102	45, 30±20	—	—	1.1°/
Carbo (Om)	87	45, 50, 70±20	< 20	—	1.0°/
Spearman (Om)	46	25±20	< 20	—	4.0°/
Grant (Of)	38	< 20	< 20	—	5.1°/
Carlton (Off)	8.7	< 20	< 20	—	4.2°/

Table IV

Ge DISTRIBUTION IN THE METALLIC PHASES
OF IRON METEORITES

Meteorite	Nichiporuk (1958)		This Study	
	Kamacite	Taenite	Kamacite	Taenite
Canyon Diablo	23	2200	220±40	200–430
Toluca	28	450	170±20	170–380

ILLUSTRATIONS