An Investigation of Equilibrium Diagram of Fe-As-C System

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Introduction

The present investigation was carried out in order to obtain the Fe-As and Fe-As-C equilibrium diagrams ranging from 0 to 40.16% arsenic and from 0 to 6.67% carbon as the fundamental research for the influence of arsenic upon the properties of steel.

Preparation of Samples

Armco iron, Swedish carbon steel, Kenjiho white pig and metallic arsenic, their composition being shown in Table 1, were charged as raw materials into an alundum-lined graphite crucible and melted in a Tammann furnace. The molten alloy weighing 100 or 150 gr. was cast into a cast iron or sand mould preheated to about 500°C. Shapes of samples are shown in Fig. 1, where (A) and

Table 1. Chemical Analyses of Raw Materials

Materials	Composition (%)					
	C	Si	Mn	P	S	Cu
Armco iron	0.016	0.030	0.04	0.003	0.011	0.035
Swedish carbon steel	0.090	0.25	0.33	0.010	0.021	—
"	0.32	0.24	0.38	0.013	0.035	—
"	0.42	0.28	0.40	0.013	0.026	—
"	0.49	0.30	0.40	0.026	0.009	—
"	0.71	0.26	0.24	0.025	0.014	—
"	0.90	0.30	0.22	0.025	0.009	—
"	1.12	0.29	0.23	0.023	0.014	—
"	1.31	0.31	0.25	0.024	0.020	—
Kenjiho white pig	4.01	0.005	tr.	0.028	0.008	0.061
Metallic arsenic	As=99.9					

(B) show the cast forms, (a) and (b) illustrate the dimensions of the specimens used for the measurement by the differential dilatometer and the differential thermal analysis respectively.

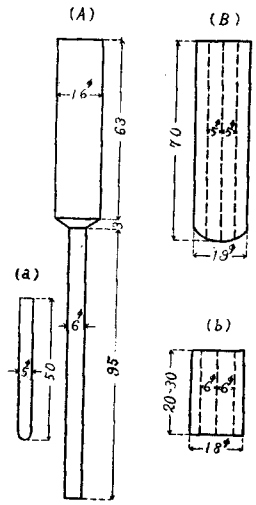


Fig. 1. *Shapes of samples.*

Experimental Method

A small rod of specimen illustrated in Fig. 1 (a) and a neutral body made of Fe-Ni (Ni=25%) alloy, its size being the same as the specimen, were set in the Uno-type differential thermal dilatometer which had been partly improved. The annealing temperature and time applied for the specimens are shown in Table 2 in

Table 2. Annealing Method

Marks of Specimens				Annealing Method
0.01.5 0.13.0 0.15.0 0.17.0 0.00.0 0.70.0 0.00.3 1.10.3 0.50.6 1.30.6 0.71.0 0.315 0.313	0.08.0 a 0.13.5 0.16.0 0.18.0 a 0.10.0 a 0.90.0 0.30.3 1.30.3 0.70.6 0.11.0 a 0.91.0 0.615 0.613	0.010 a 0.14.0 0.16.5 0.110 0.30.0 1.10.0 0.50.3 0.00.6 0.90.6 0.31.0 1.11.0 0.311	0.12.0 0.14.2 0.16.7 0.115, 0.50.0, 1.30.0, 0.90.3, 0.30.6, 1.10.6, 0.51.0, 1.31.0, 0.611,	900°C. for 2 hr. ⟶ Furnace cooling ⟶ 370°C. ⟶ Air cooling. (Kept in lead bath above 370°C. and air cooled below 370°C.)
0.32.5 0.92.5 1.72.5 0.37.5	0.52.5 1.12.5 2.02.5	0.72.5 1.32.5 0.07.5	0.82.5 1.52.5 0.27.5	1°C./min. 900°C. for 3 hr. ⟶⟶ 730°C. ⟶ Furnace cooling. (In atmospheric nitrogen gas.)
0.57.5 0.957.5	0.77.5 1.17.5	0.87.5 1.37.5	0.97.5 1.57.5	1°C./min. 840°C. for 3 hr. ⟶⟶ 690°C. ⟶ Furnace cooling. (In atmospheric nitrogen gas.)
0.01.0 a	0.02.0 a	0.02.5 a	0.09.0 a	900°C. for 5 hr. ⟶ Furnace cooling. (In atomospheric nitrogen gas.)
0.10.0 b 0.03.0 0.07.0 0.09.6	0.11.0 b 0.03.5 0.07.5 0.010 b	0.02.0 b 0.04.0 0.08.0 b	0.02.5 b 0.06.0 0.09.0 b	900°C. for 10 hr. ⟶ Furnace cooling. (In atmospheric nitrogen gas.)
0.011	0.012 b			830°C. for 20hr. ⟶ Furnace cooling. (In atmospheric nitrogen gas.)
0.01.0 c	0.02.0 c	0.02.5 c		1,200°C. for 1 hr. ⟶ Thermal analysis was performed beginning with this temperature.

which the mark of sample "0.12.0", for example, means that the specimen was made so as to contain 0.1% carbon and 2.0% arsenic. The heating and cooling velocity through the critical range were 1°C./1~5 min., and the measurements were carried out in atmospheric nitrogen gas. The apparatus for differential thermal analysis is shown in Fig. 2. The weight of the specimen for this analysis was ca. 40 gr. and the measurements were carried out in atmospheric nitrogen gas at the cooling velocity of 1~2°C./min.. The main part of the apparatus for quenching is shown in Fig. 3. As shown in the figure, the specimen was hung by a nichrome wire to keep it at the center of the silica tube. After keeping it for 30 min. at the required temperature, by turning the upper cock, the specimen was dropped within 0.3 sec. into ice water together with the nichrome wire. This quenching method was adopted mainly for the purpose of determining the γ-loop range and the solidus line at the section containing 0.14% carbon.

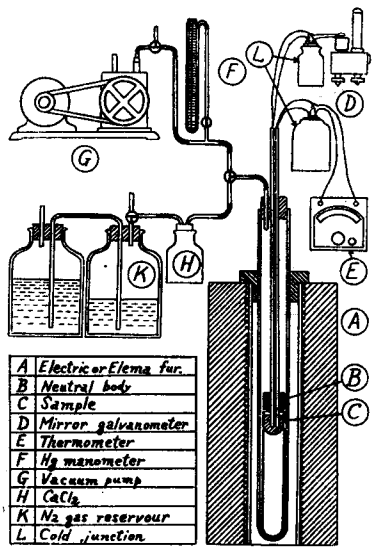


Fig. 2. *Apparatus for differential thermal analysis.*

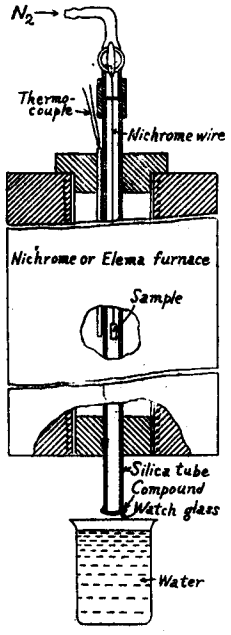


Fig. 3. *Main part of apparatus for quenching.*

Experimental Results

The microscopic structure of the specimens, having been used for the measurement by the differential dilatometer two or three times after the annealing at 900°C. for 10 hr. or 830°C. for 20 hr. in atmospheric nitrogen gas, was examined to determine the solubility of Fe_2As in ferrite, and we could

find that there remained only ferrite in the specimens which contained less than 9.69% arsenic and the minimum amount of Fe_2As in the specimen containing 11.35% arsenic. We could hardly decide whether the compound Fe_2As existed or not in the specimen containing 10.03% arsenic.

In order to determine the solubility of Fe_2As in ferrite or in austenite in the Fe-As-C system, eighteen kinds of the specimens which contained respectively 9, 10 or 11% arsenic and 0.1, 0.3, 0.6, 0.9, 1.2 or 1.5% carbon were annealed as follows:

Annealing (A): 850°C. for 10 hr. $\xrightarrow[\text{slow cooling}]{1°C./2\ min.}$ 810°C. for 3 hr. $\xrightarrow[\text{slow cooling}]{1°C./2\ min.}$ furnace cooling below 770°C.

Annealing (B): 850°C. for 20 hr. $\xrightarrow[\text{slow cooling}]{1°C./2\ min.}$ 810°C. for 3 hr. $\xrightarrow[\text{slow cooling}]{1°C./2\ min.}$ furnace cooling below 770°C.

By the microscopic examination of these specimens, the existence of Fe_2As was confirmed as shown in Table 3, in which the symbol "A" and "B" correspond to the annealing methods (A) and (B), the symbol "+" indicates the existence of Fe_2As, the symbol "−" indicates its non-existence and the symbol "±" indicates that it is difficult to determine whether the compound exists or not.

Table 3. Results of Microscopic Examination

%C \ %As	9	10	11	Annealing
0.1	−	−	+	A
	−	−	±	B
0.3	−	−	+	A
	−	−	+	B
0.6	−	−	++	A
	−	−	++	B
0.9	−	+	++	A
	−	±	++	B
1.2	−	+	++	A
	−	+	++	B
1.5	+	++	++	A
	+	++	++	B

The microscopic structure of four specimens, annealed at 785°C. for 30 hr. and furnace cooled, the composition of which are respectively (3.40% C, 3.35% As), (3.46% C, 4.30% As), (3.40% C, 5.31% As) and (3.26% C, 6.04% As), showed that (1) Fe_2As had disappeared in the 1 st and 2 nd specimens, (2) a very small amount of it had remained in the last specimen, and (3) the confirmation of the existence of the compound was impossible in the 3 rd specimen.

The typical microscopic structures of binary and ternary alloys are shown in Photographs 1~14.

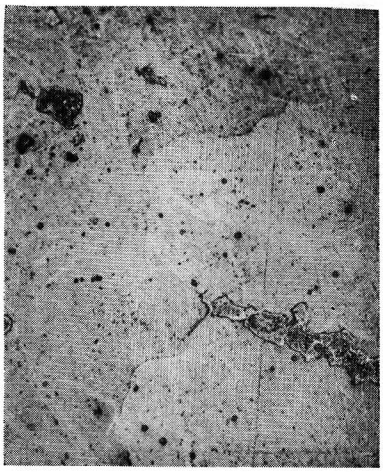


Photo. 1 ×300
C=0.16%, As=3.31%
900°C.×2hr. F.C. 5% Picral etch.
Ferrite+Pearlite.

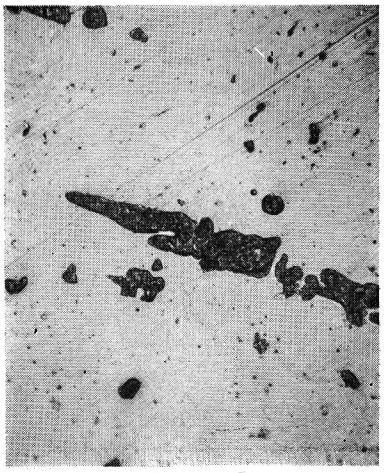


Photo. 2 ×300
C=0.14%, As=8.31%
800°C.×30 min. W.Q. 5% Picral etch.
Ferrite+Martensite.

Photo. 3 ×450
C=0.14%, As=1.09%
986°C.×30 min. W.Q. 5% Picral etch.
Martensite.

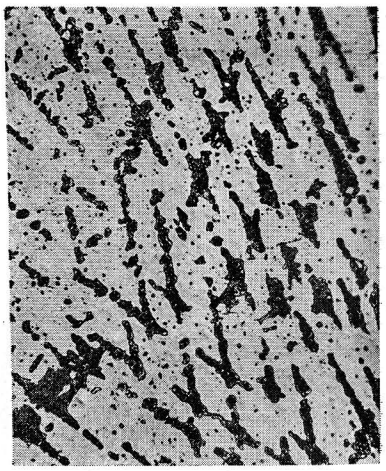


Photo. 4 ×100
C=0.20%, As=6.43%
880°C.×30 min. W.Q. 5% Picral etch.
Ferrite+Martensite.
(Pearlite partly remains.)

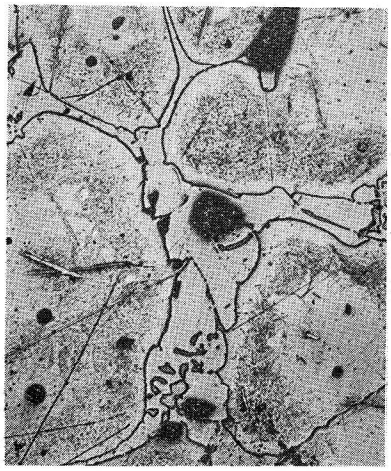


Photo. 5 ×100

C=0.048%, As=12.18%

Slowly cooled structure, 5% Picral etch.
Ferrite Dendrite + (α-Fe + Fe_2As) Eutectic.
(Acicular pearlite-cementite is found in ferrite grain. Black spots are holes.)

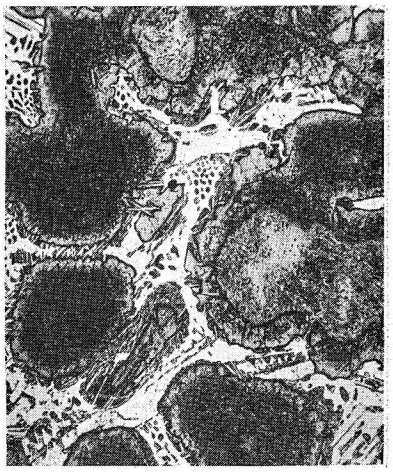


Photo. 6 ×100

C=0.22%, As=11.85%

Slowly cooled structure, 5% Picral etch.
(Ferrite+Austenite) Dendrite+Peritecto-eutectic+Pearlite in Dendrite.
(Austenite transforms to ferrite below 826°C.)

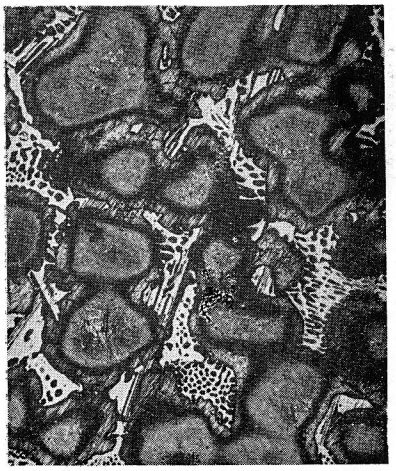


Photo. 7 ×100

C=0.55%, As=11.68%

Slowly cooled structure, 5% Picral etch.
Austenite Dendrite + (γ-Fe + Fe_2As) Eutectic+Pearlite in Dendrite.
(Austenite transforms to ferrite below 810°C.)

Photo. 8 ×100

C=1.51%, As=12.46%

Slowly cooled structure, 5% Picral etch.
Austenite Dendrite + (γ-Fe + Fe_3C) Eutectic+Ternary Eutectic+Pearlite in Dendrite+Graphite.
(Austenite transforms to ferrite at 800°C.)

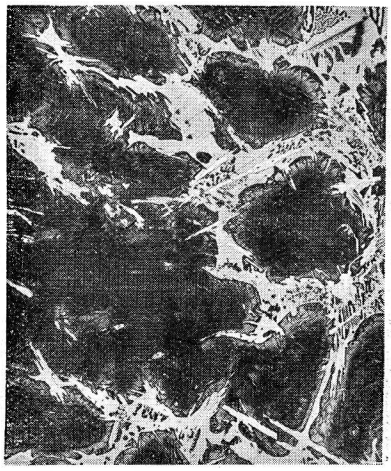


Photo. 9 ×100

C=2.59%, As=12.05%

Slowly cooled structure, 5% Picral etch.
Austenite Dendrite + (γ-Fe + Fe_3C) Eutectic + Ternary Eutectic + Pearlite in Dendrite+Graphite.
(Austenite transforms to ferrite at 800°C.)

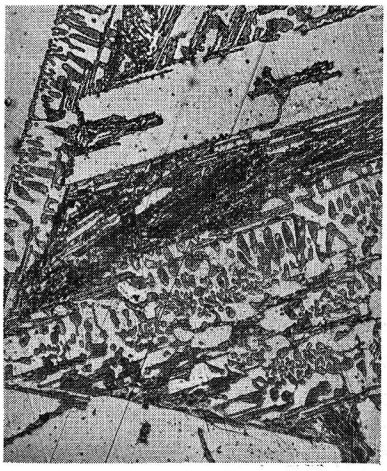


Photo. 10 ×100

C=0.30%, As=30.14%

Slowly cooled structure, 10% Ferricyanide alkaline sol. etch.
Primary Fe_2As+(γ-Fe+Fe_2As)Eutectic +Ternary Eutectic.

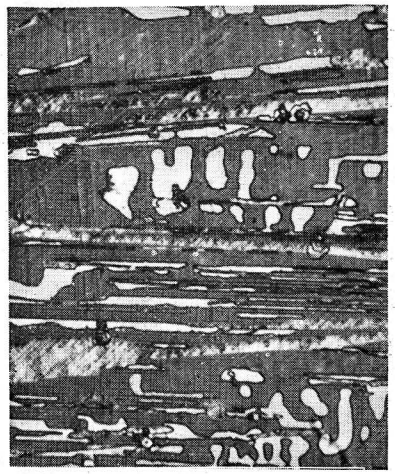


Photo. 11 ×600

C=0.83%, As=25.4%

Slowly cooled structure, 10% Ferricyanide alkaline sol. etch.
Austenite i.e. Ferrite below 800°C. (white)+Fe_3C (mottle, green)+Fe_2As (matrix, violet).
(The portion of ternary eutectic).

Photo. 12 ×300

C=0.030%, As=36.38%

Slowly cooled structure, 5% Picral etch.
Fe_2As Grain+(α-Fe+Fe_2As) Eutectic.
(Eutectic Fe_2As coagulates to Fe_2As grain.)

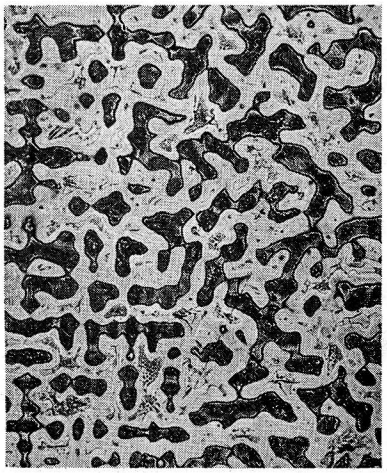


Photo. 13 ×100
C=0.50%, As=10.05%
Cast structure, 5% Picral etch.
Austenite Dendrite + (γ-Fe + Fe_2As)
Eutectic+Ternary Eutectic+Pearlite
in Ferrite Grain.

Photo. 14 ×300
C=0.50%, As=10.05%
Annealed structure, 5% Picral etch.
Ferrite+Pearlite.
(Pearlite-cementite is partly spheroidized.)

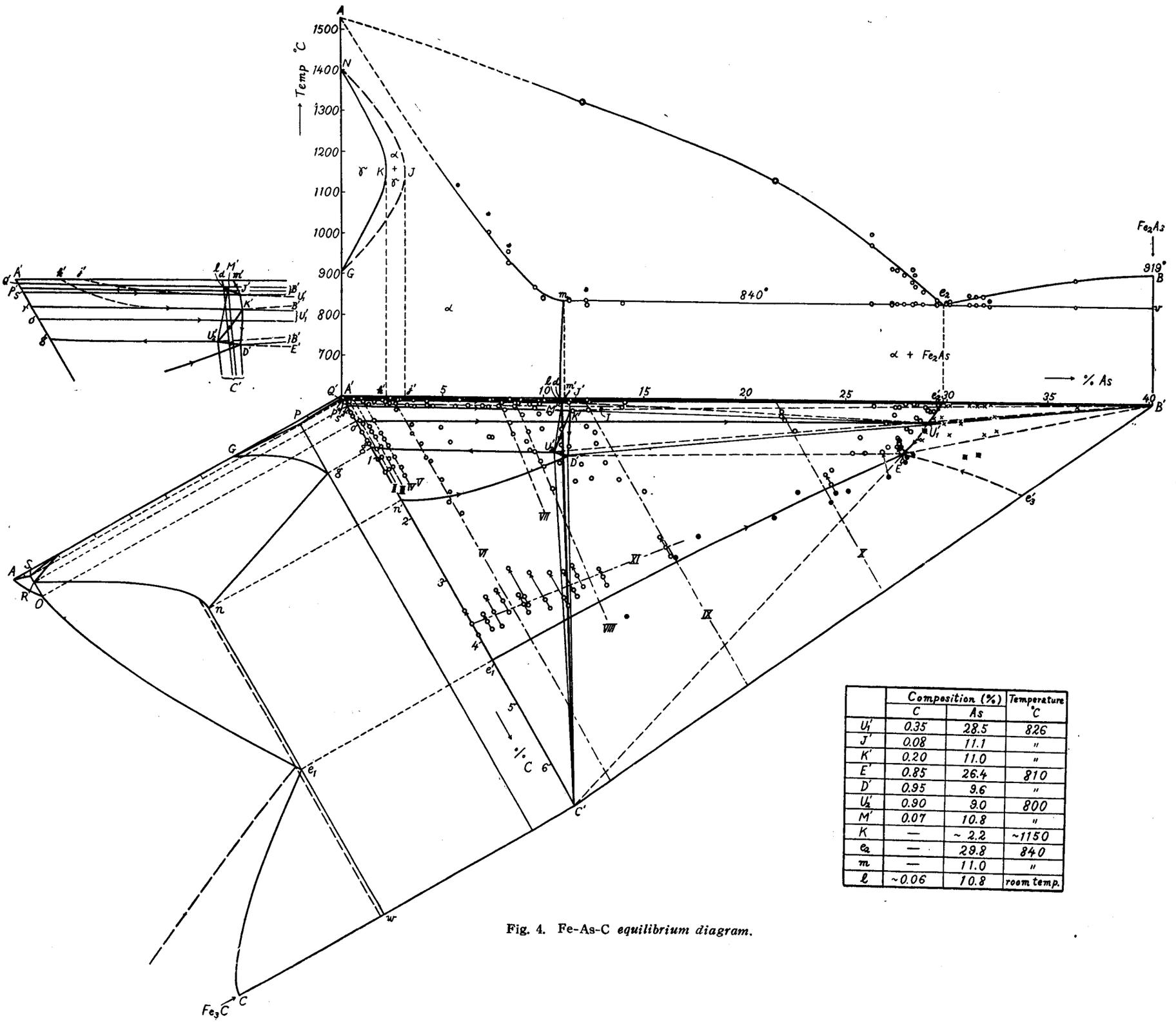
Photo. 1~14: Area reduced 27% for reproduction.

By summarizing these results, together with those obtained by the differential thermal analyses and with the recordings of the differential dilatometer, the Fe-As equilibrium diagram and eleven sections of the Fe-As-C system were obtained as shown in Figs. 4~16. The symbol "◎" in Figs. 4, 5, 6, 11 and 13 indicates the correspondent points when any one of these sections is compared with another section. The symbols "●" and "○" indicate the transformation points during heating and cooling respectively. The broken lines in Fig. 12 show the phase change, assuming that the results obtained in the section I are accurate.

Considerations

1. Fe-As System.

There are three main experimental data reported by K. Friedrich,[1] P. Oberhoffer and A. Gallaschik[2] and G. Hägg[3] concerning the Fe-As equilibrium diagram. M. Hansen[4] proposed a diagram, shown in Fig. 17, where the phase relation between ferrite and austenite is drawn as a closed γ-field according to the point of view of F. Wever.[5] E. Jänecke[6] gave another kind of diagram, illustrated in Fig. 18, in which ferrite is produced at ca. 960°C from austenite and melts by a peritectic reaction, thus attaching importance to the data given by P. Oberhoffer and A. Gallaschik.



	Composition (%)		Temperature °C
	C	As	
U_1'	0.35	28.5	826
J'	0.08	11.1	"
K'	0.20	11.0	"
E'	0.85	26.4	810
D'	0.95	9.6	"
U_2'	0.90	9.0	800
M'	0.07	10.8	"
K	—	~ 2.2	~1150
e_2	—	29.8	840
m	—	11.0	"
l	~0.06	10.8	room temp.

Fig. 4. Fe-As-C *equilibrium diagram.*

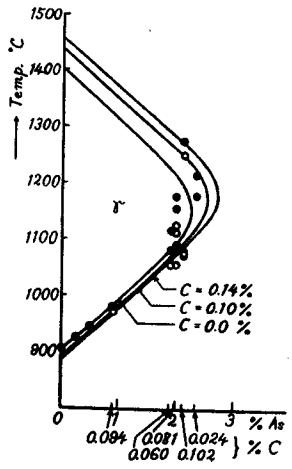


Fig. 5. *Shapes of γ-loops.*

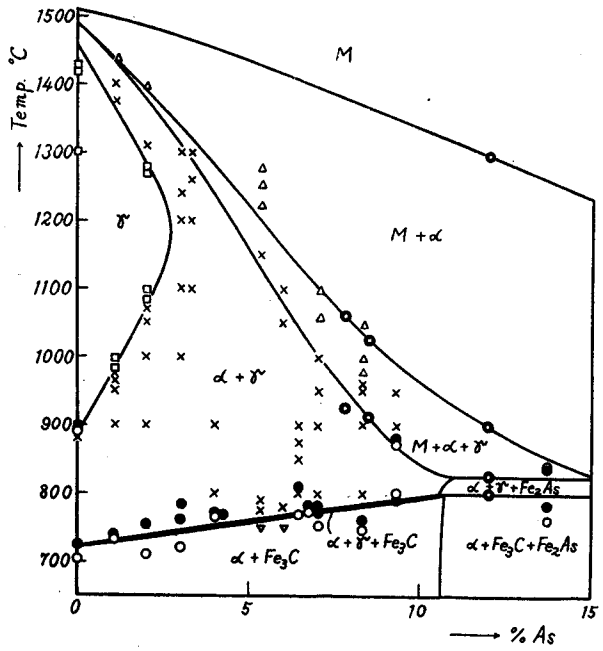


Fig. 6. *Section* I (C=0.14%).

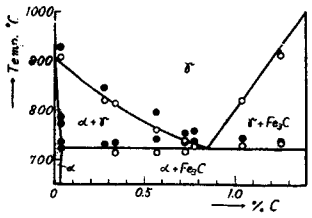


Fig. 7. *Section* II (As=0.0%).

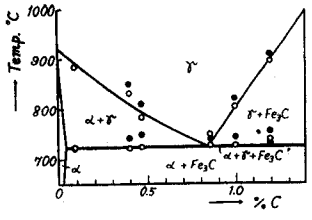


Fig. 8. *Section* III (As=0.25%).

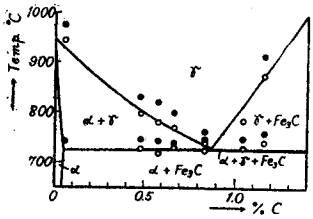


Fig. 9. *Section* IV (As=0.5%).

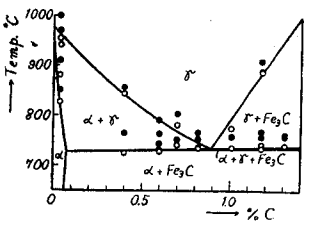


Fig. 10. *Section* V (As=0.9%).

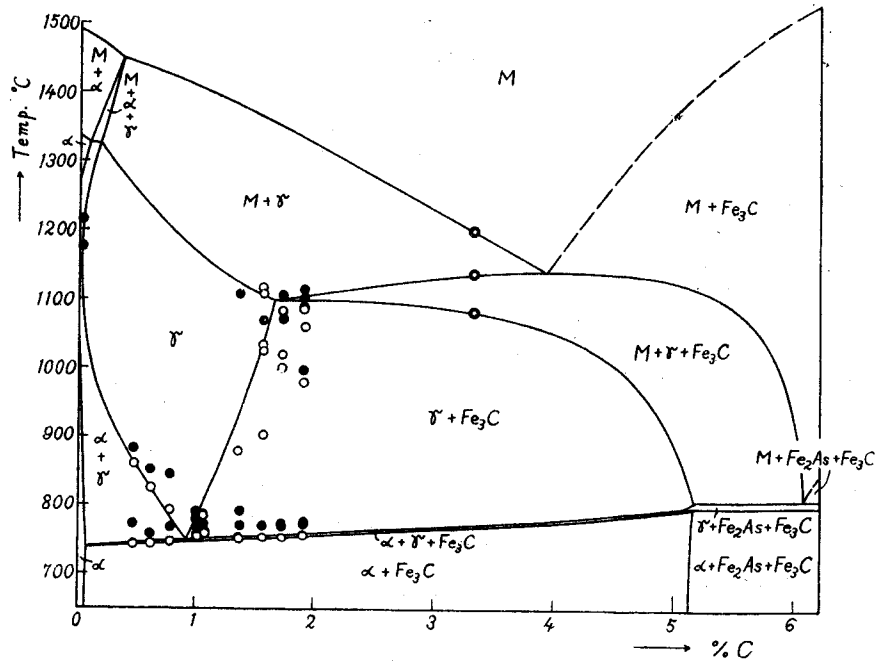


Fig. 11. *Section* VI (As=2.5%).

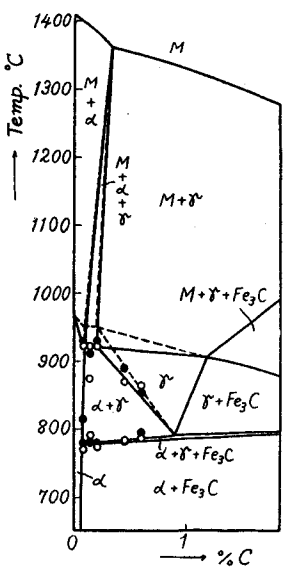


Fig. 12. *Section* VII (As≅7.5%).

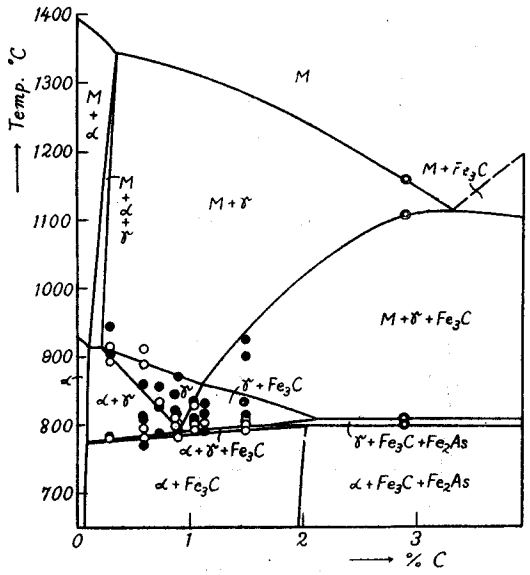


Fig. 13. *Section* VIII (As≅8.5%).

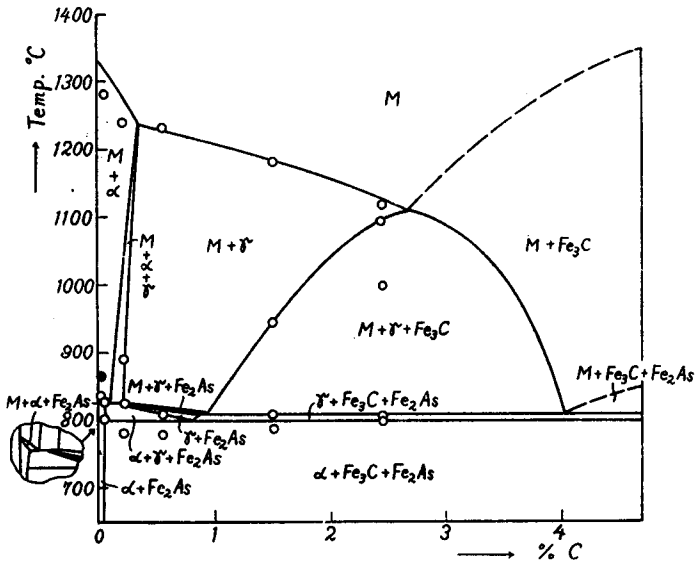


Fig. 14. *Section* IX (As=12%).

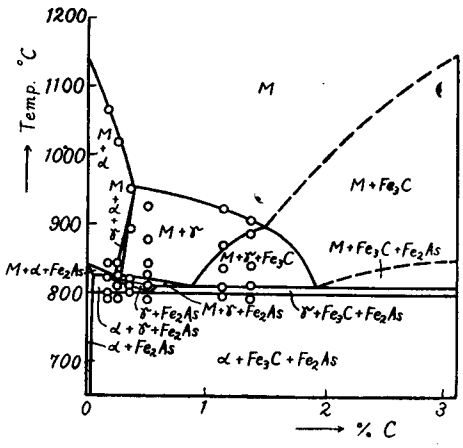


Fig. 15. *Section* X (As=21.5%).

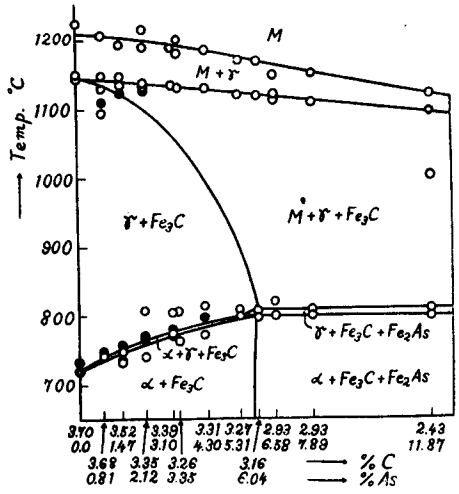


Fig. 16. *Section* XI.

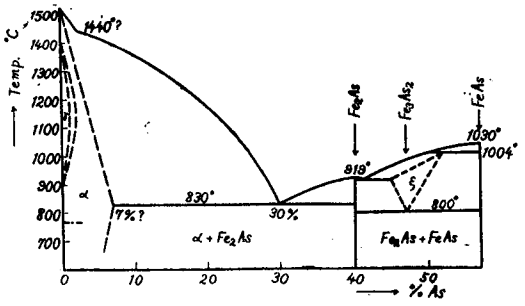


Fig. 17. Fe-As (M. Hansen).

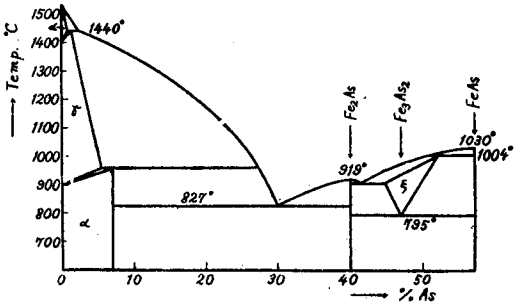


Fig. 18. Fe-As (E. Jänecke).

We could not obtain the specimens with small enough carbon content to determine accurately the γ-loop of the binary system, because the melting of these specimens was carried out in the reducing gas atmosphere. Therefore, we determined the γ-loop in the section containing 0.14% carbon by examining the microscopic structures of the quenched specimens. The maximum solubility of arsenic in austenite in this section was found to be approximately 2.7% at 1,180°C. as shown in Fig. 5. The γ-loop was not clearly determined from the results of observations of the specimens in the section containing 0.10% carbon, but no transformation point could be observed between the range from 1,000 to 1,300°C. in the specimen containing 0.053% carbon and 2.68% arsenic. The maximum solubility of arsenic in austenite was determined to be 2.6% at 1,170°C. judging from these results as shown in Fig. 5. By extrapolating these two loops to the Fe-As system, the maximum solubility of arsenic in austenite in this binary system was determined to be about 2.2% at 1,150°C. which is lower than the value decided by W. D. Johnes.[7]

The solubility of ferrite in austenite, i.e. the outer γ-loop, could not be determined because this curve spreads out rapidly as the content of carbon becomes higher and reaches to the curve "s'J'M'p'" at the content of 0.07 or 0.08% carbon.

The solubility of Fe_2As in ferrite is 10.8% arsenic at room temperature and it becomes 11.0% at 840°C.

The peritectic reaction at the vicinity of 960°C. proposed by E. Jänecke was not observed. The liquidus lines coincide nearly to those given by K. Friedrich.

We could not, however, measure the temperatures of the liquidus line ranging from 0 to 10% arsenic because it lies higher than 1,300°C. and our apparatus was unavailable for use in the range above this temperature. The composition and the temperature of eutectic point "e_2" are as follows:

$$e_2: \ As = 29.8\% \quad \text{at } 840°C.$$

2. Fe-As-C System.

In order to obtain the diagram of the Fe-As-C system, we determined many sectional diagrams of this system based on the Fe-As system obtained above, and the metastable Fe-C diagram given by F. Körber and H. Schottky.[8]

We found that the figure of the ternary system is quite similar to the Fe-P-C system reported by R. Vogel.[9]

There is no new ternary compound within the range of Fe-Fe_2As (As=40.16%)-Fe_3C (C=6.67%). Liquidus planes are as follows:

Primary ferrite crystallizes out of the liquidus plane "A′o′U_1′e_2′",
„ austenite „ „ „ „ "o′e_1′E′U_1′",
„ cementite „ „ „ „ "e_1′C′e_3′E′",
„ Fe_2As „ „ „ „ "B′e_2′U_1′E′e_3′".

"U_1′" is the peritecto-eutectic point and "E′" is the ternary eutectic point. The reactions at these two points are as follows:

$$\alpha_{J'} + (\text{melts})_{U_1'} \rightleftarrows \gamma_{K'} + Fe_2As \quad \text{at } 826^\circ C.,$$
$$(\text{melts})_{E'} \rightleftarrows \gamma_{D'} + Fe_3C + Fe_2As \quad \text{at } 810^\circ C.$$

The specimens which contain primary austenite or ferrite are shown by the symbol "○" in the ternary diagram, the specimens containing primary Fe_2As are shown by the symbol "×" and those shown by the symbol "●" contain kish graphite. The specimen shown by the symbol "⊠" contains only a binary or ternary eutectic and no primary crystal. The reason why we could not obtain the specimen composed only of ternary eutectic after the thermal analysis, in spite of many trials, is the fact that the content of carbon decreased or the kish graphite floated out of the specimen during cooling. Several points connected by the arrow at the vicinity of ternary eutectic "E′" show the specimens, the composition of which changed during the thermal analysis.

Because of the separation of kish graphite, we could not determine the eutectic line of Fe_3C and Fe_2As, but the eutectic line may be a curve "e_3′E′" judging from the shapes of the planes "e_1′C′e_3′E′" and "B′e_2′U_1′E′e_3′".

The relative relation between ferrite and austenite in the portion of the range where the open and closed γ-field are connected is somewhat complicated and is shown, therefore, by the ideal diagram drawn at the left side of the ternary diagram. The eutectoid point "g′" rises gradually with the increase of arsenic and it reaches the peritecto-eutectic point "U_2′". The phase change in the peritecto-eutectoid plane is as follows:

$$\gamma_{U_2'} + Fe_2As \rightleftarrows \alpha_{M'} + Fe_3C \quad \text{at } 800^\circ C.$$

As the content of arsenic increases, pearlite-cementite has the tendency to grow in the acicular form, and this tendency is remarkable, particularly in the specimens containing more than 7% arsenic and the low amount of carbon.

The two sections shown in Figs. 12 and 13 are more inaccurate than the other sections and we could not decide, therefore, the planes "J′M′U_2′K′" and "K′D′U_2′" by theses results alone. We examined further the microscopic structure of many long time annealed specimens described on page 132 and were able to determine the positions "J′, K′, D′, U_2′ and M′". A_3-line rises gradually

as the content of arsenic increases, but A_{cm}-line scarcely changes its temperature though the content of arsenic increases.

3. Composition Change of Specimens.

The specimens were analysed but the results are not shown in this paper except some examples.

In the melting of specimens containing carbon more than the amount indicated at "e_1'E'" and "E'B'", kish graphite was produced, and, at the same time, the content of arsenic showed a remarkable decrease though the reason is uncertain. The composition of the specimens containing less than 1.5% carbon and 12% arsenic did not change even after long time annealing. Some examples of them are shown in Table 4. We had to make suitable corrections in the specimen values used for the study of section XI which showed considerable difference in the content of carbon and no change in the content of arsenic after the thermal analysis; the three points connected by the arrow show the composition change where the middle point illustrates the composition adopted as shown in Table 5.

Some of the specimens contained an unexpected high amount of silicon and the reason was found to be in the use of Swedish carbon steel as raw materials.

Therefore, Armco iron, Kenjiho white pig and metallic arsenic were used for the preparation of specimens thereafter. A_1- and A_3-transformation points of the specimens belonging to the sections II, III, IV and V seem to be somewhat higher than those of the specimens containing no silicon.

Table 4. Some Examples of Change in Composition

Marks of Specimens	Composition (%)			
	C		As	
	As Cast	After Thermal Analysis	As Cast	After Thermal Analysis
0.011	0.025	—	11.3	11.3*, 11.4※
1.37.5	1.12	1.15†	8.11	—
0.926	0.87	0.74	26.9	26.3
1.026	0.98	0.73	26.3	26.3
1.027	0.91	0.67	26.4	26.5
1.229	1.23	0.83	24.9	25.4

* Annealed at 830°C. for 20 hr.

※ Annealed at 830°C. for 20 hr. and then used for the two successive measurements by dilatometric method.

† Annealed at 840°C. for 3hr. and then used for the measurement by dilatometric method.

Table 5. Change in Composition

Marks of Specimens	Composition (%)				
	C			As	
	As Cast	After Thermal Analysis	Mean Value	As Cast	After Thermal Analysis
3.90.0	3.89	3.50	3.70	0.0	—
3.80.8	3.80	3.55	3.68	0.81	—
3.71.6	3.75	3.28	3.52	1.47	1.57
3.62.5	3.52	3.17	3.35	2.12	—
3.63.5	3.40	2.84*	3.26	3.35	—
3.54.2	3.46	2.87*	3.31	4.30	—
3.53.3	3.53	3.24	3.39	3.10	3.36
3.45.0	3.40	2.87*	3.27	5.31	5.25
3.35.8	3.26	2.85*	3.16	6.04	—
3.26.6	3.09	2.76	2.93	6.58	—
3.17.5	3.05	2.80	2.93	7.89	—
2.512	2.58	2.27	2.43	11.87	—
1.522	1.50	1.20	1.35	21.90	—

* Thermal-analysed and then annealed at 785°C. for 30 hr.

Summary

(1) Nearly 200 kinds of specimens containing iron, arsenic and carbon were made and differential thermal analysis, direct thermal analysis, measurement by the differential dilatometer and microscopic examination of the long time annealed or quenched specimens were carried out. From these results the Fe-As binary diagram and eleven sections of the Fe-As-C ternary system, as shown below, were determined.

1. Fe-As system (As=0~40.16%),
2. Figure of the γ-loop,
3. Section I (C=0.14%, As=0~14%),
4. „ II (C=0~1.26%, As=0.0%),
5. „ III (C=0~1.20%, As=0.25%),
6. „ IV (C=0~1.16%, As=0.5%),
7. „ V (C=0~1.31%, As=0.9%),
8. Section VI (C=0~1.90%, As=2.5%),
9. „ VII (C=0~0.61%, As≅7.5%),
10. „ VIII (C=0~1.49%, As≅8.5%),
11. „ IX (C=0~2.59%, As=12%),
12. „ X (C=0~1.50%, As=21.5%),
12. „ XI ({C =3.70%, As=0.0%} ~ {C =2.43%, As=11.87%}).

(2) The Fe-As-C equilibrium diagram was obtained by summarizing these results. The compositions and the temperatures of the invariant points of the ternary system are as follows:

	%C	%As	Temp. °C.		%C	%As	Temp. °C.
U_1'	0.35	28.5	826	M′	0.07	10.8	800
J′	0.08	11.1	„	K	—	≅2.2	1,150
K′	0.20	11.0	„	e_2'	—	29.8	840
E′	0.85	26.4	810	m	—	11.0	„
D′	0.95	9.6	„	1	≅0.06	10.8	room temp.
U_2'	0.90	9.0	800				

(3) The eutectic line of Fe_3C and Fe_2As and the outer γ-loop of the Fe-As system could not be determined.

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