1907

Chemical and physical analysis of steel and iron

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Recommended Citation

Ericson, John T. E., "Chemical and physical analysis of steel and iron" (1907). *Bachelors Theses*. 275.
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THESIS

FOR THE

Degree of Bachelor of Science

IN

Metallurgical Engineering.

JOHN T. E. ERICSON.

SUBJECT:

"Chemical and Physical Analysis of Steel and Iron."

JUNE, 1907.

DETERMINATION OF SILICON.

Samples were taken as follows:

Steel A.	1 = 3.0015 gr drillings 11= 3.0026 " "
Iron B	1 = 2.0036 gr " 11 = 2.0029 " "
Steel C	1 = 5.0048 gr " 11 = 5.0046 " "

Each sample was dissolved in 40 cc nitric acid of 1.2 specific gravity and evaporated to dryness in air bath and towards end temperature was raised so as to decompose ferric nitrate.

The evaporating dish was removed from airbath and allowed to cool. Then 30 cc hydrocgloric acid was added and heat gradually increased until the ferric oxide was completely dissolved.

The dish was again placed in the airbath and solution evaporated to dryness,then the residue was redissolved in 30 cc hydrochloric acid and this diluted to 150 cc by water and filtered.

The evaporating dish was thoroughly cleaned with a "policeman" and the contents poured upon the filter which had previously been washed with dilute hydrochloric acid. Washings were continued until chlorine could not be testified in the filtrate.

Found the residue on the filter not white enough to be accepted for pure silica, so I fused it six times its weight of sodium carbonate,dissolved in water, acidulated with hydrochloric acid, evaporated to dryness,redissolved in hydrochloric acid and water,diluted,filtered,washed,ignited and weighed.

Obtained percentage of Silicon as follows:

Sample A

No.1 Final weight of crucible = 23.3871

Original weight of " = 23.3823

SiO_2 = .0051

$$\text{Percentage of Si} = \frac{.0051 \times .4702 \times 100}{3.0015} = .07946$$

No.2. Final weight of crucible = 22.7157

Original " " " = 22.7105

SiO_2 .0052

$$\text{Percentage of Si} = \frac{.0052 \times .4702 \times 100}{3.0026} = .08139$$

Sample B

Had to fuse the final residue here.

No.1

Final Weight of crucible = 13.7433

Original " " " = 13.7004

SiO_2 .0429

$$\text{Percentage of Si} = \frac{.0429 \times .4702 \times 100}{2.0036} = 1:-$$

No.2. Final Weight of Crucible = 11.4746

Original " " " = 11.4322

SiO_2 = .0424

$$\text{Percentage of Si} = \frac{.0424 \times .4702 \times 100}{2.0029} = .9903$$

Sample C.

Used the H.F. here.

No. 1

weight of crucible before H.F. = 23.3909

" " " after H.F. = 23.3792

SiO_2 = .0117

$$\text{Percentage of Si} = \frac{.0117 \times .4702 \times 100}{5.0048} = .109\%$$

No. 2. Had to fuse here the silicous residue on account of being not white enough.

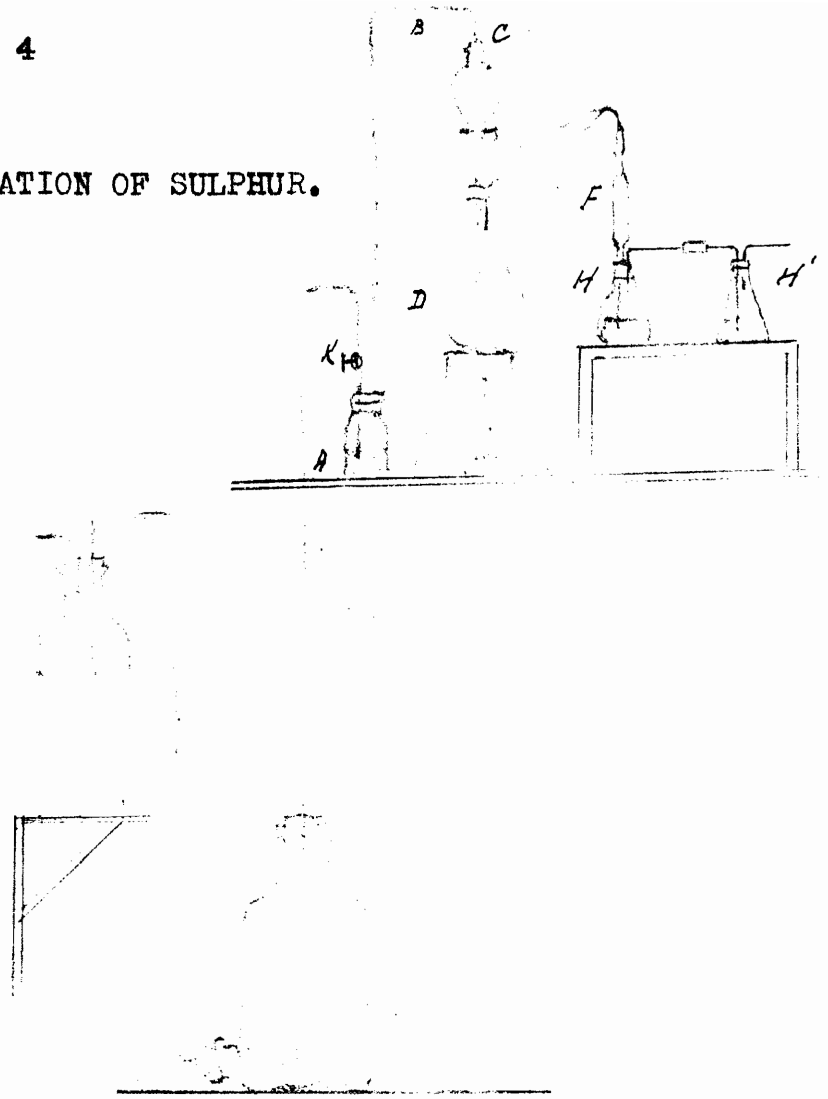
Final weight of crucible = 22.7207

Original " " " = 22.7080

SiO_2 .0127

$$\text{Percentage of Si} = \frac{.0127 \times .4702 \times 100}{5\overset{5}{.}0048} = .119\%$$

METHOD IN DETERMINATION OF SULPHUR.



The diagram above shows the arrangement for carrying out the process. The wash bottle A contained an alkaline solution of lead nitrate and was connected with the funnel tube by the rubber tube B, and a small piece of glass tubing, C, turned down at a right angle with one and drawn down and covered with a short piece of fubber tubing. This was fitted in the neck of the bulb of the funnel tube and made a tight joint.

The analytical process was conducted as follows:

I took a five factor weight of each sample (6.878 grams), which was perfectly free from lumps and placed the drillings in a previously dried flask D, and closed it with a rubber stopper fitted with a funnel tube. The outlet tube from the flask D connects with the tube F, reaching almost to the bottom of the Erlemeyer flask H. Poured into each of the flasks H about 20 cc of potassium hydrate solution of lead nitrate and enough water to fill them two-thirds full.

(The lead nitrate solution used was prepared as follows: I poured a cold solution of lead nitrate into caustic potash, 1.27 specific gravity,stirred constantly to dissolve the lead oxide,which precipitated. Added the lead nitrate until a permanent precipitate was obtained. Allowed this to settle and siphoned the clear liquid into a glass stoppered bottle for use)

I connected the apparatus and ran a slow stream of hydrogen through until all the air was expelled,then closed the glass stopcock of the funnel tube and shut off the supply of hydrogen by closing the stopcock K. The connections were found perfectly tight so the liquid in tube F. did not recede. Now I disconnected the tube C, and filled the bulb with a mixture of 50 cc of strong hydrochloric acid and 50 cc of water. Replaced the tube C, turned on hydrogen and opened the stopcock of the funnel tube, so as to admit the acid into flask D. When the adid had run into flask,regulated the flow of hydrogen so that the gas could pass through the solution in the flasks H and HV as rapidly as possible and heated flask D. When solution in flask D was boiled for 15 minutes and all the metal was dissolved,removed the source of heat and continued the current of hydrogen for ten minutes,regulated its flow by means of the stopcock K, to avoid any reflux of liquid in H, which would occur if flask D was cooled down.

Hydrogen was shut off now and the apparatus disconnected and the contents of the flask H was washed carefully into a beaker. There was no precipitate of lead sulphide in flask H' so therefore its solution was used over for next sample.

The precipitate from flask H was caught on a filter and washed a few times with hot water and then while still moist the residue and filter paper was thrown into a beaker in which had been placed previously some powdered potassium chlorate and 15 cc strong hydrochloric acid. This was allowed to stand in a moderately warm place until the fumes were passed off, then I added about twice its volumne of hot water and filtered into a beaker. Washed several times with hot water,heated the filtrate to boiling and added ammonia until solution was slightly alkaline to litmus paper.Now I acidulated with a few drops of hydrochloric acid and added 15 cc of barium chloride solution and boiled 20 minutes and allowed it to stand aside for one hour,then filtered and the remaining barium sulphate on the filter of each sample resulted as follows:

(1= 6.878
Sample A (
(11= 6.878

(1 = 6.878
Sample B(
(11 = 6.878

(1 = 6.878
Sample C (
(11 = 6.878

All samples were weighed up exactly the same.

Sample A

Took 6.878 drillings

No.1 Final weight of crucible = 23.3985

Original " " " = 23.3880

$BaSO_4$ = .0105

$$\text{Percentage of S} = \frac{.0105 \text{ X } .1375 \text{ X } 100}{6.878} = .0209\%$$

No. 2. Took 6.878 drillings

Final weight of crucible = 22.7316

Original " " " = 22.7212

$BaSO_4$ .0104

$$\text{Percentage of S} = \frac{.0104 \text{ X } 1375 \text{ X } 100}{6.878} = .02\%$$

Sample B

Took 6.878 gr. drillings

No. 1

Final weight of crucible = 22.7318

Original " " " = 22.7118

$BaSO_4$ .0200

$$\text{Percentage of S} = \frac{.0200 \text{ X } .1375 \text{ X } 100}{6.878} = .39\%$$

No. 2 Took 6.878 drillings

Final weight of crucible = 23.3998

Original weight of " = 23.3797

$BaSO_4$ = .0201

$$\text{Percentage of S} = \frac{.0201 \text{ X } .1375 \text{ X } 100}{6.878} = 0401\%$$

Sample C.

Took 6.878 to each

No.1

Final weight = 22.7364

Original " = 22.7210

$BaSO_4$ .0154

$$\text{Percentage of S} = \frac{.0154 \times 13.75}{6.878} = .0307\ \%$$

No. 2

Took 6.878 gr drillings

Final weight = 23.4185

Original " = 23.4030

$BaSO_4$ .0155

$$\text{Percentage of S} = \frac{.0155 \times 13.75}{6.878} = .0309\%$$

Had to rum several samples until the apparatus given could give me a fair result.

METHOD IN DETERMINATION OF PHOSPHOROUS.

Reagents

Nitric acid of	1.135 sp.gr.
Strong Sulphuric Acid	1.84 " "
Dilute Sulphuric Acid	2 1/2%, by volumne,made by diluting 25 cc of concentrated C.P. sulphuric acid to one litre of distilled water.
Strong Ammonia	.90 sp.gr.
Dilute "	.96 " "

Strong solution of Potassium permanganate,for oxidizing the phophorous and carbonaceous matter in the nitric acid solution of steel. It was made by dissolving 15 gr. of crystallized potassium permanganate in one litre of distilled water and then filtered through asbestos.

Standard solution of potassium permanganate,for titrating the reduced solution of ammonium potassium permanganate in one litre of distilled water and filtering through asbestos

An attempt was made to make use of the reductor but a good reductor was not at hand so the method of process was carried out as follows:

Amount of dample as specified below was weighed up and transferred to an Erlenmeyer flask,poured into the flask 100 cc of nitric acid)1.135 sp.gr) and covered with a watch glass. Heated until the solution was complete and nitric oxide expelled.

Added 10 cc of strong potassium permanganate solution,boiled,until the pink color disappeared and manganese dioxide separated. Continued the boiling for several minutes,then removed the beaker from the heat and added a small crystal of ferrous sulphate and repeated it until the manganese dioxide was completely dissolved. Boiled now two minutes longer,placed the flask in a dish of cold water and being cool I poured 40 cc of dilute ammonia (.96 sp.gr.) into the beaker. The precipitated ferric hydrate was redissolved when liquid was perfectly mixed. When solution was at a temperature of my hand, I added 40 cc of molybdate solution,closed the flask with a rubber stopper and shook it for five minutes by hand. Then allowed the precipitate to settle for few minutes, filtered and washed with acid ammonium sulphate solution until 3 cc of the wash water gave no reaction for molybdium with a drop of ammonium sulphide. Poured 5 cc of ammonia (.90 sp.gr.) and 20 cc of water into the flask to dissolve any adhering ammonium phospho molybdate and then poured it on the precipitate in the filter. Allowed the filtrate to run into a flask. Washed most carefully. Now I added to the solution five grams of pulverized zinc and poured it in in such a way so as to not allow any zinc to cling to side of beaker. Now 15 cc of strong sulphuric acid (1.84 sp.gr.) was added. Closed the flask with a rubber stopper carrying a glass tube bent twice at 90 degrees,the long arm dipping into a beaker containing a saturated solution of sodium bicarbonate.

The flask was allowed to stand undisturbed for 1/2 hour. Solution had a greenish color and titration was done with a previously done blank also.

Results of Standardization of Permanganate Solution and the process of treatment of the sample are as follows:

Took

No. (1) .1011 Met. Fe required 16.6 cc

)2) .1041 " " " 17.1 "

Wire used contained 99.87% Fe.

$$1 \text{ cc perm. solu.} = \frac{.1011 \times .9987}{16.6} = 006080 \text{ gr.Fe.}$$

Ratio of Molybdic acid to iron = .85714

Ratio of phosphorous to molyb. acid = .01794

Sample A

No. (1) 1.0056 gr. steel required 2.3 cc pot. perm. solu.

No. (2) 1.0080 " " " 2.4 " " " "

Therefore

$$\text{No. (1)} \quad \frac{2.3 \times .006080 \times .85714 \times .01794 \times 100}{1.0056} = .021\% \text{ P.}$$

$$\text{" (2)} \quad \frac{2.4 \times .006080 \times .85714 \times .01794 \times 100}{1.0080} = .022\% \text{ P.}$$

Sample B.

1.0079 gr drillings required 8.6 cc pot. perm. solu.

1.0030 " " " 8.5 cc " " "

Therefore as .006080 X .85714 X .01794 = .00009349

$$\text{No. (1)} = \frac{8.6 \times .00009349 \times 100}{1.0079} = .079\ \% \text{ P.}$$

$$\text{No. (2)} \quad \frac{8.^{5} \times .00009349 \times 100}{1.0030} = .079\% \text{ P.}$$

Sample C.

1.0004 gr steel took 3.3 cc of pot. perm. solu.

1.0014 " " " 3.3 " " " " "

No. (1) $$\frac{3.3 \text{ X } .00009349 \text{ X } 100}{1.0004} = .03\% \text{ P.}$$

No. (2) $$\frac{3.3 \text{ X } .00009349 \text{ X } 100}{1.0004} = .03\% \text{ P}$$

Blanks were run and taken care of.

METHOD IN DETERMINATION OF MANGANESE.

Reagents:

Nitric acid	(sp.gr.1.135)
Nitric acid	3 per cent.
Permanganate solution	1 gr. of
salt to liter of water.	
Ferrous Ammonium Sulphate solution	12.4 gr.
Strong Sulphuric Acid	50 cc to
one litre of water.	

The strength of ferrous sulphate solution was continually tested by the following way:

Measured into a 200 cc flask 50 cc of nitric acid (sp.gr.1.135) cooled, and added a very small amount of bismuthate, diluted with 50 cc of three per cent nitric acid, filtered into a 300 cc flask and washed with 50 cc of 3% nitric acid.

Ran into solution from a pipette 25 cc of ferrous sulphate solution and titrated with potassium permanganate until faint pink.

The permanganate solution was standardized in the usual way and results given below:

Weighed up amounts of each sample as noted below. The sample was dissolved in 50cc of nitric acid (sp.gr.1.135) in an Erlemeyer flask. Cooled and added 1/2 gram of bismuthate. Heated for a few minutes until the pink color was gone. Added now sufficient amount of ferrous, sulphate solution to clear the solution of the sample and heated until all nitrous oxide was driven off.

Cooled to about 15 degrees C, added an excess of bismuthate and agitated for a few minutes. Added not 50 cc of water containing 30 cc of nitric acid to the litre or 3%, and then filtered through an asbestos felt on a platinum cone into a 300 cc Erlenmeyer flask and washed with 3% nitric acid solution. Ran now into the flask from a pipette a measured volumne of ferrous sulphate solution and titrated to a faint pink color with permanganate with following results:

Standardization of the Pot. permanganate solution.

(1) .1044 gr Fe req. 65.2 cc of solution

(2) .1034 " " " 64.7 " " "

Found the strength of the ferrous am. sulphate solution to be to pot. permanganate solution as 20.1 cc pot. permanganate solution was found to be equivalent to 25 cc of ferrous ammonium sulphate solution.

Found also that 1 cc of potasium permanganate solution would oxidize $\frac{.1044}{65.2}$ = .001601 gr. Met. Fe.

Therefore 1 cc of solution is equivalent to .00160 X .294 = .000470694 Manganese.

Took sample of drillings as follows:

A (1= 1.0004 gr
(11= 1.0012 "

B (1= 2.0032 "
(11= 2.0031 "

C ((1) = 1.0046 gr
((2) = 1.0048 "

Having performed the process as directed above and having added a measured volumne of ferrous ammonium sulphate solution to do away at exact point, the pink color that was formed in test solution, then I began to titrate the test solution until faint pink color was obtained.

A (Sample (1) required .8 cc perm. solu.
(" (2) " .9 " " "

B (Sample (1) " .9 " " "
(" (2) " .9 " " "

C (Sample (1) " .6 " " "
) " (2) " .6 " " "

Consequently in deducting the number of cubic centimeters of potassium permanganate solution obtained from the number corresponding to the volumne of ferrous sulphate solution used obtained results as follows:

A ((1) = $\frac{.000470694 \text{ X } 19.2 \text{ X } 100}{1.0004} = .903\%$ Mn.
((2) $\frac{.000470694 \text{ X } 19.3 \text{ X } 100}{1.0012} = .907\%$ "

B ((1) $\frac{.000470694 \text{ X } 19.2 \text{ X } 100}{2.0032} = .45\%$ "
((2) $\frac{.000470694 \text{ X } 19.2 \text{ X } 100}{2} = .45\%$ "

$$C \left(\begin{array}{l} \dfrac{.000470694 \times 19.5 \times 100}{1.0046} = .913\ \%\ \text{Mn.} \\ \\ \dfrac{.000470694 \times 19.5 \times 100}{1.0048} = .913\ \%\ \text{Mn.} \end{array} \right.$$

Blanks were also run and taken care of.

METHOD OF DETERMINATION OF TOTAL CARBON.

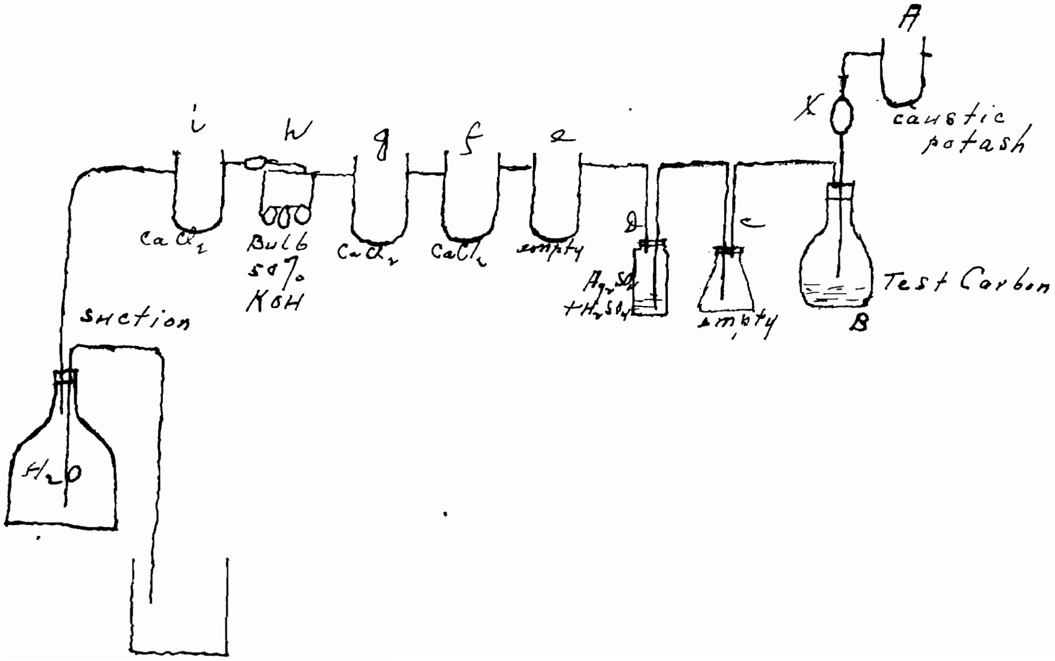
Specified amount of each sample below was weighed up and dissolved in a solution of 200 cc of double chloride of Copper and Ammonium and 15 cc of strong hydrochloric acid.

The solution was frequently stirred and allowed to stand three days. Now it was filtered on a platinum cone with asbestos which previously dried and burned so as to be free of any carbonaceous matter.

The residue was first washed with dilute hydrochloric acid and then several times with hot water.

Filter pumps with suction was used in filtration.

After the carbonaceous residue was thoroughly washed it was gently dried and carefully transferred to a 700 cc flask.



Above diagram represents the outfit for process. In the U tube A, we find caustic postash. Into bulb X I poured 10 cc of saturated solution of chromic acid followed by 200 cc strong sulphuric acid containing a few cc of hot chromic acid. This was allowed to pour down very slowly into bottle or flask B, containing the carbonaceous residue free from water. Flask C. is empty and serves as condenser. Bottle D contains a solution of Silver sulphate in strong sulphuric acid. The first U tube E is empty while the two U tubes F and G are filled with dry calcium chloride.

Bulb H contains a 50% solution to absorb the carbonic acid This bulb is carefully detached and weighed after operation is finished.

The last U tube contains also dry calcium chloride. The air was drawn through by means of suction.

METHOD OF DETERMINATION OF GRAPHITE.

I dissolved the weighed sample in Nitric acid sp.gr.1.2, using 15 cc of acid to each gramme taken for analysis.

Filtered on an asbestos filter perfectly free from carbonaceous matters.

The residue was transferred carefully upon the filter and thoroughly washed with hot water and then I treated the residue with hot caustic potash solution 1.1 sp.gr. and then again washed thoroughly with hot water, then with dilute hydrochloric acid and finally with hot water.

Having obtained the residue perfectly washed and free from water and dry, it was transferred to the bottle and the carbon was burned exactly in same way as carbon in determination of total carbon.

Original weight of bulb	85.7711
A (1)	85.8437
A (2)	

CO_2 for A (1) = 85.8437 - 85.7711 = .0726 carbon

CO_2 " A (2) = 85.9163 - 85.8437 = .0726 "

CO_2 for C (1) = 85.9170 - 85.9163 = .0007 graphite.

Now I changed solution in bulb and proceeded again.

original wgt. = 85.4425

CO_2 for C (2) = 85.4432 graphite

CO_2 " C (1) = 85.5287 carbon total

Co_2 " C (2) = 85.6076 " "

CO_2 " A (1) = 85.6082 graphite

CO_2 " A (2) = 85.6088 "

CO_2 " B (1) = 85.8586 carbon total

CO_2 " B (2) = 86.1084 " "

CO_2 " B (1) = 86.4387 graphite

CO_2 " B (2) = 86.7641 "

Consequently we get.

Graphite CO_2 for C (2) = 85.4432 - 85.4425 = .0007

Total Carbon CO_2 for C (1) = 85.5287 - 85.4432 = .0855

" " " " "(2) = 85.6076 - 85.5287 = .0789

" Graphite " " A (1) = 85.6082 - 85.6076 = .0006

" " " " " (2) = 85.6088 - 85.6082 = .0006

" Carbon " " B (1) = 85.8586 - 85.6088 = .2498

" " " " " (2) = 86.1084 - 85.8586 = .2498

" Graphite " " " (1) = 86.4338 - 86.1084 = .3254

" " " " " (2) = 86.7641 - 86.4387 = .3254

Final Calculations.

Steel A

Took (1) 2.0024 gr drill

(2) 2.0041 " "

$CO_2 : C = .0726 : X$

$44 : 12 = .0726 : X$

$X = .0198$ total C

$$(1) = \frac{.0198 \text{ X } 100}{2.0024} = .988\% \text{ Total C.}$$

$$(2) = \frac{.0198 \text{ X } 100}{2.0041} = .988\% \text{ " "}$$

Graphite Determination:

Took (1) = 5.0010 gr. drill

(2) = 5.0000 " "

$CO_2 : C = .0006 : X$

$44 : 12 = .0006 : X$

$X = .00016$

$$(1) = \frac{.00016 \text{ X } 100}{5.0010} = .0031\% \text{ C as graphite}$$

$$(2) = \frac{.00016 \text{ X } 100}{5.0000} = .0032\% \text{ C as graphite}$$

Total Carbon - Graphite = Combined Carbon.

Therefore
.988 - .0032 = .984% Combined Carbon in Steel A.

Steel C.

Took (1) 2.0078 gr drillings

(2) 2.0032 " "

(1)

$CO_2 : C = .0855 : X$

$44 : 12 = .0855 : X$

$X = .0233$ Total Carbon

$$(1) = \frac{.0233 \text{ X } 100}{2.0078} = 1.16\% \text{ Total C}$$

$CO_2 : C = .0789 : X$

$44 : 12 = .0789 : X$

$X = .0215$ Total C.

$$(2) = \frac{.0215 \text{ X } 100}{2.0032} = 1.073 \% \text{ Total C.}$$

Graphite Determination

Took (1) = 5.0000 gr drillings

(2) = 5.0045 " "

$CO_2 : C = .0007 : X$

$44 : 12 = .0007 : X$

$X = .00019$ Carbon as graphite.

$$(1) = \frac{.00019 \text{ X } 100}{5.0000} = .0038 \% \text{ Carbon as Graphite}$$

$$(2) = \frac{.00019 \text{ X } 100}{5.0045} = .0037 \% \text{ Carbon as Graphite}$$

Therefore average = .00375 % C.

Average Total C = Av. Graphite = Av. Combined Carbon

Consequently

1.1165 - .00375 = 1.11275 % Combined Carbon.

Iron B.

Took

(1) = 2.0040 gr. drill

(2) = 2.0046 " "

$CO_2 : C = .2498 : X$

$44 : 12 = .2498 : X$

$X = .0704$ Total C in (1)

$$(1) = \frac{.0704 \times 100}{2.0040} = 3.51\% \text{ Total Carbon}$$

$$(2) = \frac{.0704 \times 100}{2.0046} = 3.51\% \text{ Total Carbon}$$

Graphite Determination

Took (1) = 3.0030 gr. drillings

(2) = 3.0021 " "

$CO_2 : C = .3254 : X$

$44 : 12 = .3254 : X$

$X = .0887$ Carbon as graphite

$$(1) = \frac{.0887 \times 100}{3.0030} = 2.95 \% \text{ C as graphite}$$

$$(2) = \frac{.0887 \times 100}{3.0021} = 2.95 \% \text{ C as graphite}$$

Total Carbon - Graphite = Combined Carbon.

Consequently

3.51 - 2.95 = .56% Combined Carbon.

METHOD IN DETERMINATION OF TITANIUM.

This is a very tedious process to operate and requires time and great care. I had to run several samples before I conquered the difficulties and obtained satisfactory results.

Blair advises to follow the process of determination of phosphorous to begin with and that is as follows:

The weighed sample was treated with 80 cc nitric acid 1.2 sp.gr. and when the violent action had ceased,added 10 cc hydrochloric acid. Evaporated the solution to dryness in the air bath, replaced the air cover,heated until the ferric nitrate was nearly all decomposed,cooled and added 30 cc hydrochloric acid,heated gradually until the iron oxide was dissolved and evaporated to dryness again in the air bath. Cooled,dissolved in 30 cc hydrochloric acid,diluted and filtered and saved residue for further treatment which shall be noted further below.

Blair's Acctate Method: He heats the filtered acid solution nearly to boil,remove beaker from light and adds gradually from a small beaker a mixture of 10 cc acid ammonium sulphite and 20 cc ammonia,stirring constantly. The precipitate first formed,redissolved and when all but about two or three cc of acid am. sulphite solution has been added, replaces the beaker over light If the precipitate on addition of am. sulphite does not dissolve by stirring he adds a few drops of hydrochloric acid and when solution clears,continue the addition,very slowly,of acid am. sulphite. After replacing the beaker on light, add to the solution ammonia,drop by drop, until the solution is quite decolorized and until finally a slight greenish precipitate remains undissolved even after stir. Now he adds the remaining 3 cc of acid am. Sulphite which throws down a white precipitate which redissolves. One thing to be noted is if the solution of acid am. sulphite is too weak then the ferric chloride will not be reduced and solution would not be decolorized and will not give a smell of sulphurous anhydride.

In that case Blair advises, of course, to add more acid am. sulphite (without the addition of ammonia) until solution smells strongly of sulphurous anhydride then we add ammonia, until a slight permanent precipitate appears, and then redissolve in a few drops of hydrochloric acid. Now the solution is very nearly neutral, the iron is in ferrous condition and an excess of sulphurous acid present. Now in case of phosphorous he adds to the solution acid and to get complete decomposition of any excess of the adid am. sulphite. Now we boil the solution and let a stream of CO_2 pass through it and expell every trace of sulphurous anhydride, then in case of phosphorous determinations we pass a current of hydrogen sulphide through it for fifteen minutes to get the arsenic down if any. Filter and the expell H_2S by CO_2 add a little bromine water and cool solution. To the cold solution we add ammonia from a small beaker slowly and finally drop by drop with stirring. The green precipitate of ferrous hydrate which forms first is dissolved by stirring. One thing to be noted is although the green precipitate dissolves, a whitish one remains and the next drop of ammonia increases the white precipitate or gives it a reddish tint and finally the greenish precipitate remains not dissolved and another drop of ammonia makes the whole precipitate appear green. If before this occurs

If before this occurs the precipitate does not appear decidedly red in color, then we dissolve the green precipitate by a drop of hydrochloric acid and add a little bromine water and then add ammonia as before and repeat until the reddish precipitate is obtained. Then in case of phosphorous determination we dissolve this green precipitate in a very few drops of acctic acid 1.04 sp.gr. when the precipitate remaining will be quite red in color, then add about 1 cc acctic acid and dilute the solution with boiling water. Heat to boil for one minute and filter rapidly and wash nnce with hot water. When precipitate is drained dry, pour about 15 cc of hydrochloric acid into the beaker in which the precipitation was made, warm gently, add about 10 cc of bromine water, pour this on the filter containing the precipitate. Wash out beaker once or twice and then wash filter with hot water. If acid in beaker is not sufficient to dissolve the precipitate completely, drop a little strong acid around edge of the filter before washing it with hot water. The scaly film of difficulty soluble oxide which may be found on boiling the accetate precipitate is caused by the presence of too much ammonium accetate, but if instruction above is carefully applied this does not accur.

Evaporate solution nearly to dryness to get rid of excess of hydrochloric acid, add to it a filtered solution of 10 gr of citric acid dissolved in 20 cc water, then 10 cc of magnesia mixture and enough ammonia to make solution slightly alkaline. Let beaker stand in cold water and when solution is cold add to it 1/2 its volumne of strong ammonia and stir.

When the precipitate of ammonium magnesium orthophosphate has begun to form, stop stirring, let it cool off for 10 minutes then vigerously several times at intervals and let it stand over night.

Filter on a small filter and wash with a mixture of two parts of water and one part of ammonia containing 2.5 gr. of ammonium nitrate to 100 cc. Dry,filter,ignite and wash.

REAL PROCESS IN THE DETERMINATION OF TITANIUM.

Above I gave full description of the method by which we can determine the phosphorous in iron and steel. This method I do not prefer to the Molybdate method which is in fact simpler and perhaps in a way more accurate. The object is to fully describe the acctate method for phosphor because in determination of Titanium we proceed to some extent similarly as we do in determination of phosphor but now when titanium is present we proceed as follows:

I dried and ignited the residue of graphite,etc. from the solution of the iron and burned off the carbon. Moistened this residue with cold water,added 10 drops of sulphuric acid and enough hydrochloric acid to dissolve the silica and evaporated until fumes of sulphuric anhydride were given off. While this was going on,I proceeded with the deoxelation of the filtrate above,but note this when sulphurous acid has been driven off,I did not pass hydrogen sulphide through the solution, but instead cooled it and proceeded with the acctate precipitation. Also instead of dissolving the precipitate,after washing it as specified above,I dried the filter and precipitated in the funnel. Cleaned out carefully the materials of precipitate which adhered to the sides of the beaker in which the original precipitate was made.

When the precipitate was dry,I transferred it to a small porcelain mortar and poured out the residue in it and then burned the filter paper separately and its ash was added to the residue in the mortar. Now I ground the precipitate and the small amount of ash in the mortar with 5 gr. sodium carbonate and a little sodium nitrate and then transferred to the crucible containing the residue which was treated by hydrochloric and sulphuric acid. The mortar was cleaned and so was the pestle by grinding a little more sodium carbonate which was added to the other portion in the crucible. Now I fused the whole for half an hour,cooled, dissolved the fused mass in hot water,filtered. Now I dried this residue and then transferred it to a platinum crucible,burned the filter,added its ash to the residue and fused the whole with fifteen times its weight of potassium bisulphate. The heat applied first was low and then increased gradually to red heat. The fused mass was allowed to cool,added to it 10 cc strong sulphuric acid and heated until it was perfectly liquid.

Now I poured it carefully into 400 cc cold water,added a little hydrochloric acid and 5 cc of ammonium bisulphite,filtered into a beaker,added ammonia until a permanent precipitate was formed,redissolved with few drops of hydrochloric acid,added - a filtered solution of 20 gr. of sodium accetate and 1/6 the volumne of the solution of accetic acid (1.04 sp.gr.),and heated to boiling.

The titanic acid was precipitated nicely in a flocculent way and quite free from iron. Now I boiled few minutes,allowed the titanic acid to settle,filtered,washed with hot water containing a little acctic acid,dried,ignited and weighed.

The following results were obtained:

Original Weight of Crucibles:

(1) = 22.7116

(2) = 23.3794

Took in sample (1) = 5.0028 gr. drill

" " " (2) = 5.0029 " "

(1) Weight of crucible TiO_2 = 22.7140

(2) " " " " = 23.3819

No.(1) = 22.7140 - 22.7116 = .0024 TiO_2

" (2) = 23.3819 - 23.3794 = .0025 TiO_2

$TiO_2 : Ti = .0024 : X$

$80 : 48 = .0024 : X$

$X = .00144$ Ti.

$$\text{No.(1)} = \frac{.00144 \text{ X } 100}{5.0028} = .028\% \text{ Ti}$$

$$\text{" (2)} = \frac{.00150 \text{ X } 100}{5.0029} = .031\% \text{ Ti}$$

METALLOGRAPHIC ANALYSIS
OF
STEEL A AND C.

Two steels were given with constituents as found specified in previous chemical analysis. Of each steel I took five samples which were treated as follows:

Samples No. 1 of A and C were heated to 850 degrees C and then cooled down gradually and slowly.

Sample No. 2 and 3C were cooled from 900 degrees C directly in air.

Sample No.3 A and 2 C cooled from 900 degrees C first directly in air for 1/2 minute and then held in 18 degrees C water for one second.

Sample No.4 A and C were cooled directly from 900 degrees C in 18 degrees C water.

Samples No. 5 A and C were cooled directly from 950 degrees C in ice water.

Temperature of furnace was measured by means of calorimeter and a piece of platinum. One thing to be noticed was that the temperature of furnace could not exceed 950 degrees C on account of too much heat loss in radiation through the furnace walls. Therefore it was with great difficulty metallographic studies can be done on account of not having the desired variable heat from 600 degrees C to 1500 degrees C. Still I endeavored to perform the best work possible with the things available.

It is a known fact if a piece of steel containing over .5% carbon be allowed to cool slowly from a high temperature and either quenched or not quenched, we find certain interesting phenomena taking place. To cite literature upon this subject we have found experimentally that the cooling at first proceeds at a uniformly retarded rate but when temperature of about 700 degrees C is reached there is an interruption in this regularity

Having given each piece of steel such a heat treatment as stated above I polished every one most carefully until there could not be distinguished the minutest line of scratch or rust or any other defect whatever. THe surface was smoother and brighter than a mirror. Now each piece was properly etched with a nitric acid solution, then washed and dried carefully and afterwards the polished surface was rubbed a little upon a soft piece of cloth and then ready for metallographic determination

In taking pictures I used ten minutes exposure and the objective Bo. 1/2 and eyepiece No. 2 but the pictures did not bring me a satisfactory result, therefore determined to reduce time of exposure to eight seconds which was more favorable.

As far as the microscope has been able to ascertain I have found that all steels not quenched seem to be made up of three constituents.

(1) Ferrite, which is iron free from carbon.

(2) Cementite = Fe_3C

(3) Pearlite which is a mixture made up of very thin plates alternately of ferrite and cementite in proportion of 2/3 ferrite and 1/3 cementite. It has

a pearly appearance.

By experiments I have found that when cooling a sample down in a slow and undisturbed manner from a temperature above critical point of the steel the crystalline structure is composed of grains of pearlite by cementite. Pearlite forms the meshes and cementite the net work itself. So following conclusions are made:

(1) Undisturbed cooling from an initial temperature lower than critical is not giving any crystalline structure.

(2) A slow and undisturbed cooling from a temperature above critical point produces crystallization.

(3) Critical point varies with chemical composition of the steel. Each impurity (at least Carbon and phosphorous) lowers the critical point.

Referring to liter ature connected with this work we have found upon further experiments of other kinds of steels that carbon and phosphorous and perhaps all impurities at a certain amount increased the size of the crystals forming.

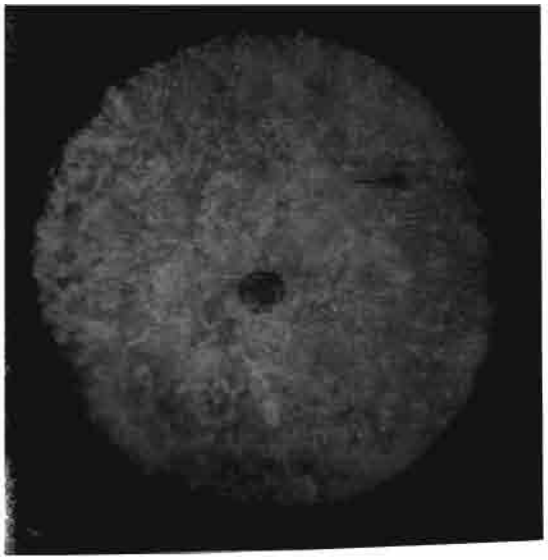
One thing of interest is this;- If we have a high class and pure steel it requires a very high temperature,at least 1000 degrees C to make any change in its crystalline structure.

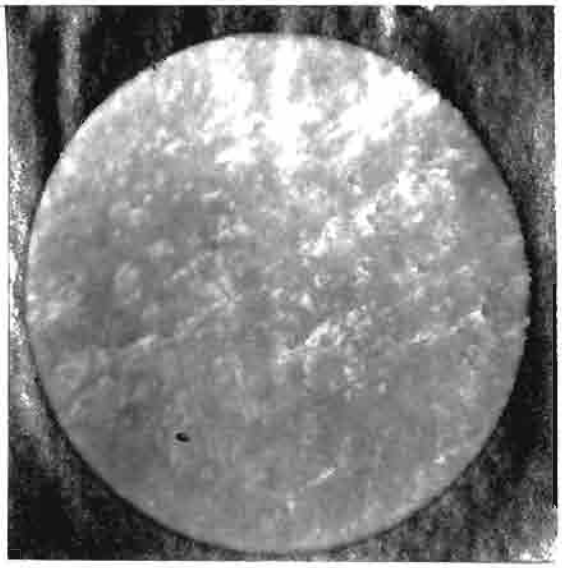
In this work pictures are inclosed but it is with difficulty we can fully express on them the very crystalline structure that was seen under the microscope.

In the samples No.1 A and C I saw the pearlite development and also in No.2A and 3C. In No.3A and 2C the pearlite was seen and ready to go into martensite.

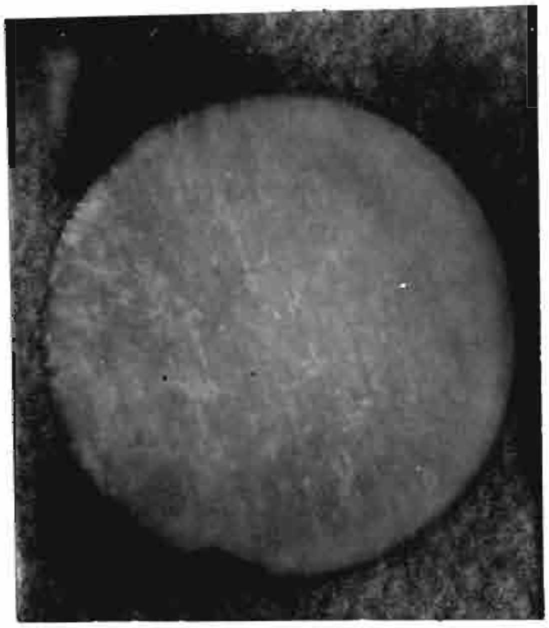
In No.4A and 4C we have martensite,which appeared under the microscope in triangular net structure intermingled with some pearlite.

Samples No.5A and 5C are also of higher structure in martensite on account of being quenched in ice water.


No 1 A


No 1 A higher power


No 2 A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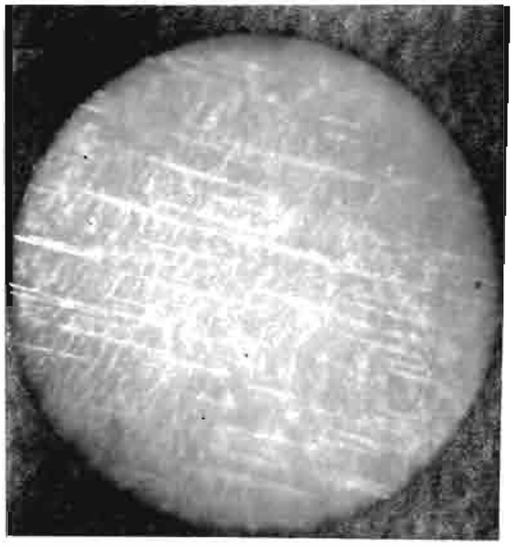
No 4 A.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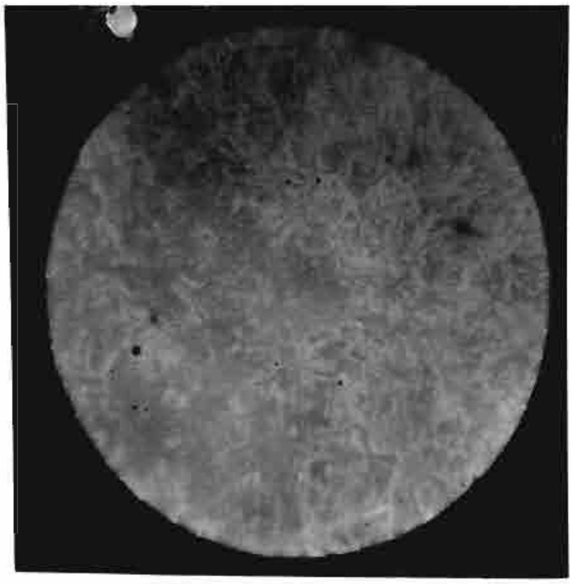
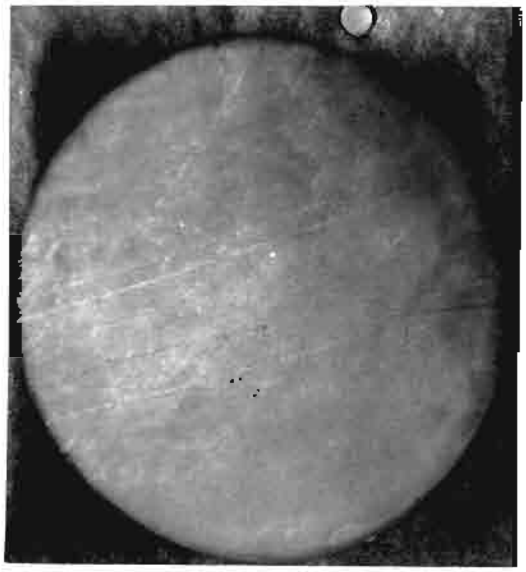


No 1 C



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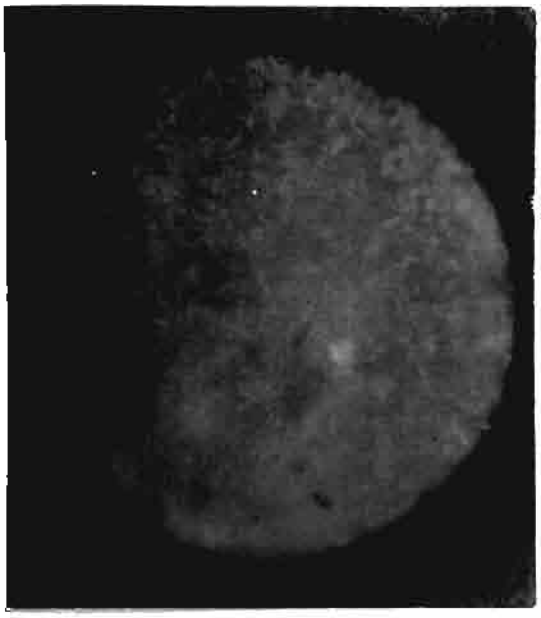
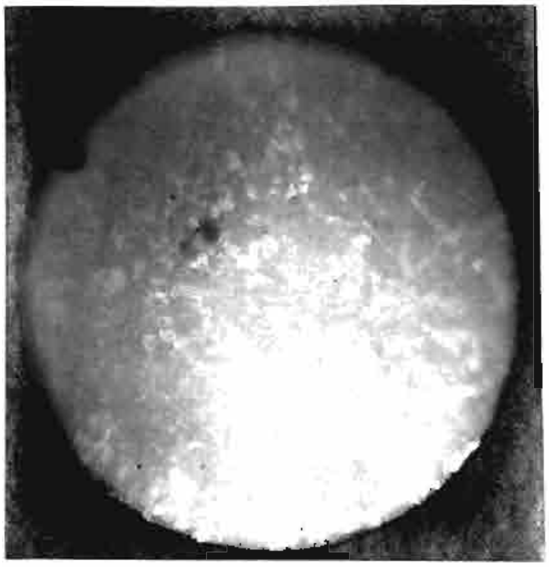
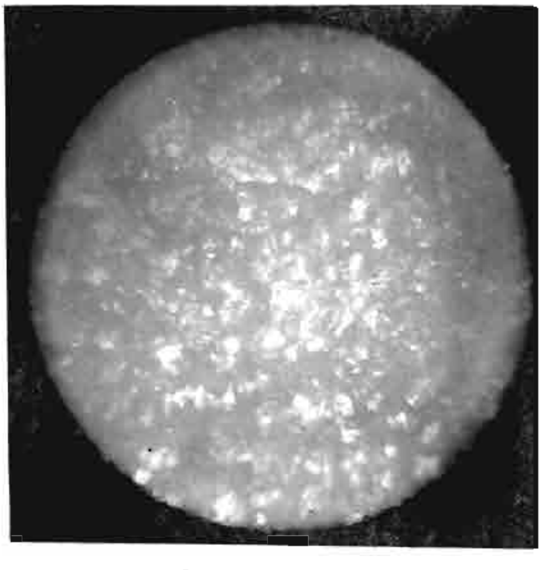


No. 3 C



No 4 C



No. 5 C