THESIS

A STUDY OF THE TRANSFORMATION POINT OF METALS

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Preface

Acknowledgment is made to:

W. G. Hilderf for designing and constructing the furnace.

H. L. Publow, Asst. Prof. of Chemistry.

The Transformation Point of Metals

This report was first started by Mr. Hilderf. The furnace was first designed and constructed by him. He however, did not have time to finish it as he wished. He managed to carry on one test with it which came out fairly good but he could not heat it up to high enough temperature because the heating coil was placed too far from the sample which was to be tested and observed. The main problem that comes up in this experiment is to keep the metal from being oxidized while being heated up.

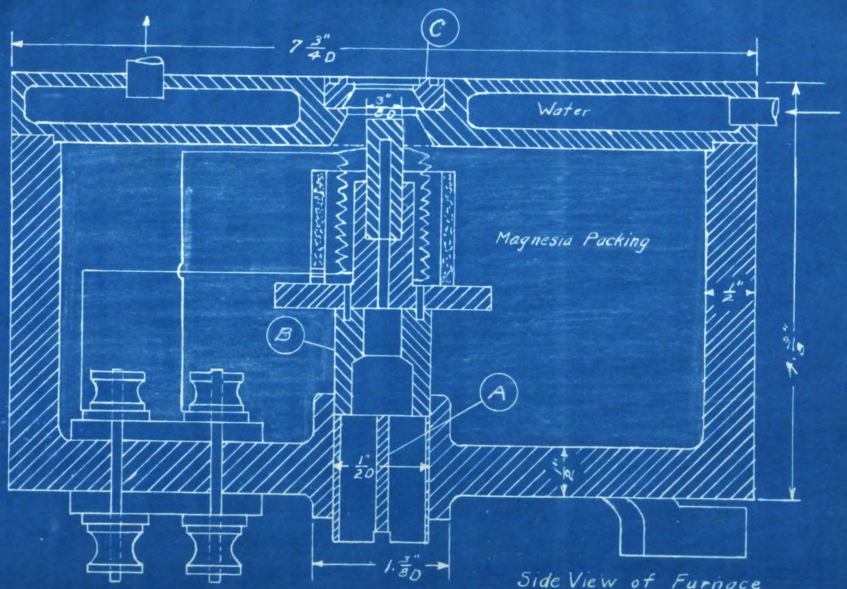
The furnace as constructed is in the form of a cylinder about 7 3/4 inches in diameter and standing about 5 inches high from the table. There is a cover to this furnace, with a hole bored thru the center and thru which the metal is observed with the microscope. This cover is hollow and has an inlet and an outlet for water which is used for keeping the microscope cool and also making it more comfortable for the operator or the observer. The hole in the center of the cover is tapped and has a brass plug which is threaded and placed in this tapped hole and may be removed at will with a special wrench. This plug has a 3/4 inch hole drilled in it and there is a piece of selected mica placed in this hole and cemented down with alundum cement. In the base of the furnace there are two holes drilled for the leads to the heating coil. The leads are, of course insulated from the casing of the furnace. The thermal couple comes into the furnace

thru a hole in the center of the bottom of the furnace. This hole is about an inch in diameter and is threaded and fitted with an inch plug. This plug is drilled with two holes about 3/8 inch in diameter. These holes are for the thermal couple. The thermal couple projected up thru this plug and into a wrought iron stand which holds the sample. This stand was at first only supported by the thermal couple and what little packing happened to be under it. This was one of the faults of the furnace. To give good results the sample should be about an eighth of an inch from the mica. This position also protects the microscope from the heat. In the way that the furnace was first built it was unhandy to remove the sample from the stand as it would get the stand out of position and possibly displace it so that the sample would be more than an eighth of an inch below the mica. This would cause the glass of the microscope to become heated up and possibly melt the cement that holds the glass in place. Another fault in the first construction of the furnace was, that there were three holes drilled in the bottom for letting out the oxygen. These holes were about 3/16 inch in diameter and were much too large as they let the oxygen in instead of letting it out. In the side of the furnace near the top was an inlet for nitrogen

which is to replace the oxygen and thus keep the piece from oxidizing. The cover to the furnace was fitted fairly close to the top of the furnace but not quite close enough. An oiled gasket was needed to make the top air tight.

Before I could conduct any test or experiment on the furnace I had to over-come these faults and re-arrange the furnace. The holes in the bottom were first plugged up and then I worked on the construction of a suitable heating coil which would bring the heat close enough to the sample. The old heating coil was about two inches in diameter on the inside. With this coil only about 1000 degrees Fahrenheit could be reached. The coil should give a temperature as high as 1600 degrees. The heating element in the new coil was made of nixrom wire, 20 gage. I tried two types of coils. The first one was made by winding this wire on an eighth inch mandrel. About twenty feet of the wire was wound in this way. This coil was then wound around the stand as close to the sample as possible and held in place by alumdum cement. This coil would not work because of short circuits which were almost impossible to stop. The next coil made was of wire wound on a 3/4 inch mandrel and then placed inside of a straight porcelain crucible, the bottom of which had been sawed off. The inside diameter of this was about an inch. One coil

was placed inside of the crucible and the other one was wound about the outside. Both of these coils were held in place with alundum cement. It was found that this coil worked alright. When it was tested it was found that 1700 degrees Fahrenheit could easily be reached with out the insulation around the coil and with the cover of the furnace off. A resistance of about 65 feet of the same wire was wound, and was used to keep the temperature from running up too fast and burning out the heating coil. Another plug was made to fit into the hole above the plug (A) thru which the thermal couple leads run. A hole was drilled thru the bottom for the couple. In the top of this plug were placed two dowel pins which fitted into the sample stand. These dowel pins held the sample stand rigid and in place so that the sample can easily be examined by the microscope. The sample itself was a piece of steel about 3/8 inch in diameter and about 1 1/4 inches long. There is a hole about 1 inch deep drilled in one end for the thermal couple. The other end of the sample is polished to be examined. The thermal couple was run up thru the plugs and into place and held there with alundum cement. This cement also helped to make the furnace air tight around the couple where it left the furnace.



Drawn by Watson

Side View of Furnace
Scale 1" = 1"

Next, the heating coil was set on the stand and the leads to it were attached to the brass screws passing thru the bottom of the furnace. The furnace around this coil was packed with magnesia to hold the heat in. About the coil was placed some fine carbon for taking up some of the oxygen that might otherwise go towards oxidizing the sample. An oiled gasket was made for the top of the furnace and placed on the top and then the cover of the furnace was screwed into place, making an almost air-tight connection.

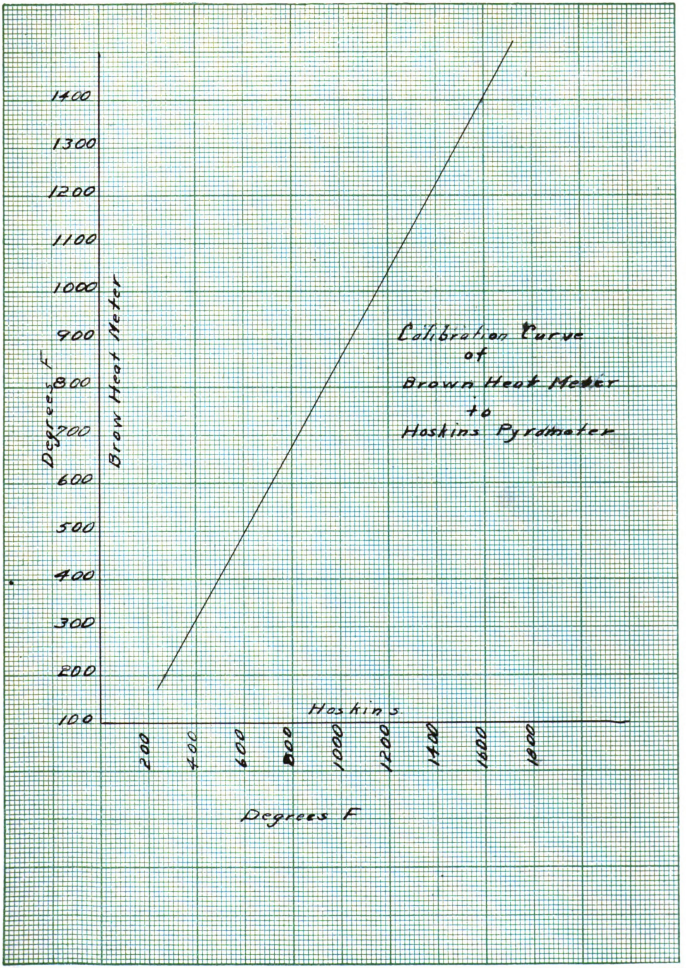
Before any test could be run on the furnace the Brown Heat Meter had to be calibrated against that thermal couple. A Hoskins pyrometer was used as a standard. Both pyrometers were connected to the same couple with a double pole double throw switch. Readins were taken on the pyrometers at the same time and then a curve was plotted. It was found that the Brown Heat Meter read about 150 degrees low. By the curve the temperature could be found from the Brown reading. The heat meter was then connected up with the couple as shown in the wiring diagram.

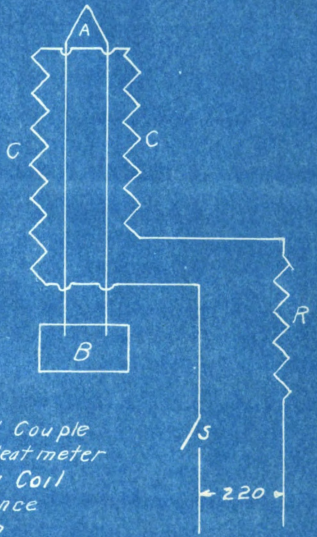
The tapped plug (C) was removed from the cover and the sample of metal to be examined was slipped into place in the stand and the cover then screwed down. Before putting this sample into the furnace, it was

Calibration of Brown Heat Meter.

Data:

Hoskins	Brown	Average Readings Hoskins	Average Readings Brown
202	I40	220	I40
310	200	310	200
400 : 400	310 : 305	400	307
530 : 530	400 : 410	530	405
600 : 600	490 : 490	600	490
700 : 700	580 : 560	700	570
800 : 800	680 : 660	750	610
900 : 900	760 : 760	800	670
960 : 950	880 : 790	900	675
I000 : I000	840 : 840	950	790
I050 : I050	890 : 880	960	820
II00 : II00	950 : 940	I000	840
II50 : II50	I000 : 980	I050	885
I200 : I200	I040 : I035	II00	945
I300 : I300	II20 : III0	II50	990
I350 : I350	II70 : II70	I200	I057
I400 : I400	I200 : I200	I250	I060
I450 : I450	I250 : I220	I300	III5
I500 :	I300 :	I350	II70
I540 :	I320 :	I400	I200
		I450	I250
		I500	I300
		I540	I320


1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
Degrees F
Brown Heat Meter
Calibration Curve
of
Brown Heat Meter
to
Hoskins Pyrometer
Hoskins
200
400
600
800
1000
1200
1400
1600
1800
Degrees F



A = Thermal Couple
B = Brown Heat meter
C = Heating Coil
R = Resistance
S = Switch

Wiring Diagram of Furnace

Drawn by C. E. Watson.

etched with nitric acid after polishing. The water was turned on so that it ran thru the cover of the furnace in the manner indicated in the figure. The microscope was set over the sample and focused and lighted. The resistance was connected in as shown in the wiring diagram and the apparatus was then ready for conducting the first test. No nitrogen or other gas was used to keep the metal from oxidizing. The current was turned on and the sample watched thru the microscope. Nothing happened until about 700 degrees and then the piece began gradually to darken over as if oxidized. The grain structure entirely disappeared and nothing could be seen on the surface of the metal. It was easily seen that this method would not work.

In the next test run on the furnace, a low carbon steel was used. In addition to this a hole was drilled in the side of the furnace and a tank of nitrogen was connected to the furnace and run into it thru this hole. The test was again repeated and with much better results as the metal did not start to oxidize till about 800 degrees. At this point there was some sort of a change in the metal. The piece or sample appeared to oxidize over entirely at about 900 degrees. At 940 degrees the structure was back in sight again and was very much more in sight than it was originally. The piece oxidized at

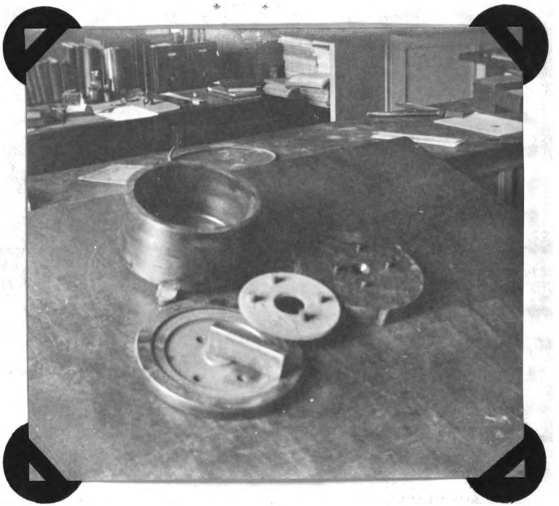
about 850 degrees without any change being noticeable.

The next test was run the same way except that a small hole about an eighth of an inch in diameter was drilled in the bottom to let out the air that was already in the furnace. Nitrogen is slightly lighter in weight than oxygen, therefore it would be almost pure nitrogen at the top of the furnace while the oxygen would gradually work out the bottom. This would leave only a small percent of oxygen in the furnace to oxidize the metal. This test came out nearly the same as the other one before. The metal appeared to oxidize at 800 degrees and then came back into sight again at 850 degrees. The metal oxidized at about 1000 degrees this time and still there was not any change to be seen in the sample.

In the third test run on the furnace the hole in the bottom of the furnace was plugged up, and then by means of a Y tube, and oil-sealed air-pump was attached to the furnace. The air was at first exhausted as far as possible and then the nitrogen was run into the furnace. The resulting mixture of nitrogen and air was then pumped out of the furnace and then more nitrogen was let in. This was repeated several times before the test was started and in this way nearly all the oxygen was taken out and replaced with nitrogen. The current was then turned on and the sample watched again.

It was found that the first change took place about 750 instead of 800 degrees as in the others. The structure disappeared again and when it came back into sight there was a darker redish color to it. There was no change till nearly 1200 and then a very slow change started. It was so slow that it was hardly noticeable, but fast enough to know that there was something going on in the sample. The ferrite or the lighter colored structure of the sample seemed to be getting smaller while the darker structure or the pearlite seemed to be getting larger all the time. This change continued till about 1300 degrees and then there did not seem to be any more change. In order to see what happened when the piece was cooled, the current was turned off. Nothing was seen because the metal immediately began to oxidize. This was probably because of the nitrogen being circulated while the sample was being heated. This nitrogen would tend to form a sort of a film about the sample which would keep the oxygen away from it. As soon as the heat was taken away the movement of nitrogen would stop and the film be broken up and let the oxygen in the furnace to the metal and thus oxidize it.

In preparation for the next test a piece of low carbon steel was obtained and turned into a standard test bar. This was pulled with a load of 10,000 pounds.



A picture of the paterns used to make the furnace and also the core box used to make the core and the core that is used in the making of the cover to the furnace.

The purpose of this is to get a piece of metal that has been stressed and see the effect of heating on it. It is said that a piece of metal that has been stressed has a large grain growth at low temperatures. If is also said that low carbon steel will not oxidize as easily as does the higher carbon steels. Some very queer things happened in this test. The piece did not exactly oxidize at the temperature the others have been doing, but it did change color. At about 750 degrees the surface of the metal to the eye appeared to be turned a deep red color. Thru the microscope the surface of the metal darkened slightly with a reddish tinge. At about 760 degrees the surface of the metal began to turn to a blue. In the microscope, the bluish color was noticeable. The light portion between the crystals turned blue and had the appearance of water. The darker portions looked like little islands in the water. On exhausting the air the metal turned back to its original color and remained there until the metal became red hot and then the metal assumed a reddish color. At 1200 degrees and a little over there was another change which took place very slowly. The surface of the sample looked very much like little globals of fat on water. The outlines of the crystals were still in sight and remained in sight till about 1300 degrees where it oxidized over. The crystals appeared to be getting a little larger but they did not grow as was expected. When the piece was taken

out of the furnace and polished and etched and then examined under a microscope it was found that the crystals had changed shape somewhat and were a little larger.

In the second test that was run on this same piece of metal nearly the same results were obtained. At about 1100 degrees another strange change took place. The crystals did not change but there was another change in the color of the sample. The surface of the metal looked very much as though a drop of water had been dropped upon it and it flowed around among the crystals. There was a bluish color which ran around among the crystals very much the same as water would do. This kept up till the entire surface of the steel was of this color. This blue color was fringed with red and purple. Nothing further was observed because at this point the mica cracked and spoiled the view of the sample.

The next test was again run on the piece of medium carbon steel. This piece again appeared to oxidize at 750 degrees and then came back into sight. Where the metal first appeared white under the microscope it now appears blue and the surface of the metal was blue to the eye also. At about 850 degrees, the metal came back to its original color again and remained there till it oxidized at about 1350 degrees. Just before it did oxidize there was a slow change in the size of the crystals.

Just before the outlines of the crystals began to disappear it was easily noticed that the crystals were growing larger. At this point the metal began to oxidize and nothing more could be seen of the change. The metal was polished and etched when it was cool and then examined under a microscope in order to make sure that there had been a change. It was found that the crystals had very nearly doubled in size. They were so large that they could be distinguished with the eye. This was the last of the tests that I ran on the two kinds of metals.

In conclusion I will say that I think better results could have been obtained if the furnace could have been made more nearly air tight. The air pump needs to be connected to the furnace with metal connections. If this had been done a higher vacuum could have been obtained. Another thing that was a source of trouble was the piece of mica which was over the sample. If too much air was exhausted from the furnace, the mica would break on account of the air pressure on the outside. Also the mica would sometimes crack and spoil the view of the sample when a high temperature was reached. A good quality of mica has to be used. A special gasket is also needed as the paper one is soon burned away and this lets air into the furnace.

Condensed Results of Tests.

Test No.	Carbon Content	Temp. F.	Appearance to eye.	Appearance Magnified 60 Diameters
I	Med.	700	Blue	Black
2	Low	800	Bluish	Black and blue
"	"	840	Bright	As originally
"	"	950	Black	Black
3	Low	800	Blue	Dark blue
2	"	850	Bright	Natural color
"	"	1000	Black	Oxidized
4	Low	750	Blue	As the oxidized
"	"	800	Bright	Natural color
"	"	1200	Bright	Grains became larger slowly.
"	"	1300	Dark	Oxidised
5	.02 C	750	Red	Had a redish tinge
"	"	760	Blue	Had watery appearance was sort of bluish
"	"	1200	Bright	Looked as globals of fat. Crystals larger
6	.02	1100	Bright	Blue red color flowed as water.
7	Med.	750	Blue	Appeared to oxidize
"	"	850	Bright	original color
"	"	1300	Bright	Grain became larger slowly.
"	"	1350	Black	Oxidised.