by

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INTRODUCTION

The exact nature and the mechanism in the formation of the various structures found in steel is a subject of much controversy.

Many methods of study have been employed to arrive at a logical explanation of the various structural changes produced in steel by different heat treating methods. These methods of study involve X-ray diffraction, microscopic examination, physical testing, change in magnetic properties and a host of others too numerous to mention.

The galvanic cell has long been used as a method of studying corrosion tendencies of various metals. Some investigations (7) show that metals heat treated to various degrees of hardness and strength will have different solubility tendencies.

It was the purpose of this study to see if a correlation between the solubility potential and physical properties of a metal--that was heat treated in various ways--could be obtained. Such a study should yield some information concerning the changes that take place in heat treatment.

THEORIES INVOLVED

The main interest in this investigation was not to prove any electrochemical theories but to apply some of these methods to the study of metallurgical problems. Therefore, the discussion of the theories involved might come under two different subheadings, first metallurgical and second electrochemical theories. However, these cannot be completely disassociated.

Metallurgy

If plain carbon steel (steel containing mainly carbon as an alloying element) is heated to above the Ac₃ temperature (6), and then cooled sufficiently fast to room temperature a hard structure known as martensite will be produced. In accordance with Sisco: (10, p. 98)

The primary cause of the hardness of martensite is the precipitation of submicroscopic particles of carbon or iron carbide from the gamma solid solution and the retention of these particles as a supersaturated solution in the tetragonal alpha-iron lattice, where they act as a multitude of keys effectively preventing slip.

Other investigators agree with Taylor (11, p. 203) in that

The martensitic constituent of a hardened carbon steel is a phase with a tetragonal crystal structure which may be looked upon as a deformation of the body centered cubic structure of alpha iron.

Statements such as the above have prompted the belief that martensite contains high internal stresses (3, p. 151).

Considering the above references, it might be concluded that the extreme hardness of martensite, as compared with other structures (sorbite, troostite or pearlite) may be due to either of the following reasons or a combination of the following: 1. a completely different lattice structure, 2. a strained or distorted lattice structure, or 3. a keying effect caused by the extremely fine particles of Fe₃C or some form of iron carbide being present. Either of the first two might be capable of causing a wide difference in electrode potential. In accordance with the findings of Cohen (1, p. 55) the strain theory was discounted. As a result of these findings, the theories become more confusing.

Electrochemical

It is a well established fact that when dissimilar metals in electrical contact with each other are exposed to an electrolyte a current flows from one to the other and is called a galvanic current. It is also accepted that a piece of metal when strained has a different electromotive force than in the unstrained condition. In accordance with Thompson (12, p. 79) if iron is strained above the elastic limit, this amounts to several hundredths of a volt, below this limit the change of electromotive force is smaller. Probably Dix and Brown (2, p. 228) have done as much work of this type as any other investigators but most of it was on aluminum alloys.

There is very little information in the literature that would directly apply to this problem from a purely metallurgical point of view. However, much data are available concerning

the problem from a corrosion standpoint. Even this information is as misleading in some instances as some of the data on metallurgical aspects. A typical example is the findings of Uhlig (14, p. 139); steel that had been quenched from above the Ac_3 temperature and then emerged in distilled or sea water corroded at a slightly higher rate than steel quenched and subsequently tempered. In 1 per cent $\rm H_2SO_4$ quenched steel (martensite corrodes the least) and with a 750° F. temper, steel corroded most. Steel tempered at 1100° F. and steel slowly cooled from the Ac_3 temperature corroded at intermediate rates.

EXPERIMENTAL PROCEDURE

Preparation of Sample

All data used in this work were obtained by using Rex AA high speed steel. This was a typical 18-4-1 type of steel. The normal chemical composition was as follows: 18 per cent tungsten, 4 per cent chromium, 1 per cent vanadium, with 0.70 per cent carbon (4) and the balance being iron.

All samples originated from a four foot, 1 x 3/8 inch annealed bar. This bar was then cut on a Doall band saw to make 3/8 inch square bars. After cutting, they were ground on a Blanchard Grinder, Model No. 11; removing about 0.017 inch of metal on all four sides. The purpose of this was to remove

any decarburized metal.

The ground samples were then cut to lengths of about 15/8 inches, marked for further identification as shown in Table 1, and heat treated accordingly.

Table 1. Identification of samples.

Samples	: Heat treatment
A1 A2 A3	Annealed condition
A4 A5 A6	Annealed condition
91 92 93	Quenched in oil from 2350° F.
21 22 23	Quenched and tempered at 200° F.
31 32 33	Quenched and tempered at 300° F.
41 42 43	Quenched and tempered at 400° F.
61 62 63	Quenched and tempered at 600° F.
81 82 83	Quenched and tempered at 800° F.
101102103	Quenched and tempered at 1035° F.
121122123	Quenched and tempered at 1200° F.
141142143	Quenched and tempered at 1400° F.

All heat treatment was accomplished by preheating to 1550° F. and then heating to 2350° F. in a Sentry Electric Furnace using carbon blocks to control the atmosphere and in this manner preventing decarburization. After heating, the samples were quenched in oil down to room temperature and then tempered in a molten salt bath at various temperatures.

After marking and heat treating, the samples were reground on the Blanchard Grinder, which was equipped with a circular magnetic chuck. To standardize the surface condition all samples were ground in the same manner and that was by having a chuck rotation of 15 revolutions per minute and a feed of 0.004 inch per minute on the wheel. This feed would give a cut of about 0.00028 inch per revolution of the chuck. The annealed samples were re-used but before each set up they were re-ground.

When the grinding operation was completed, a groove was cut about 1/16 inch from the marked end. In this indentation a 10 inch piece of 14 gage (0.064 inch) annealed copper wire was securely wrapped; thus making an electrical contact. The wire was then insulated by using plastic paint, Fig. 13. The transverse section on the lower end and the upper wired end of the sample were painted leaving about 1 1/8 inches of the sides bare.

The plastic paint used was U. S. Stoneware Company Tygon Corrosion Resistance Paint Primer, T P-107B and top coat T P-21. Each electrode was given two coats of primer and several top coats.

Cell Set Up

The galvanic cell consisted of two electrodes, the standard or reference electrode being an annealed sample; the other being a heat treated sample. These electrodes were immersed in an electrolyte consisting of 1N H2SO4 with an addition of 2.044

grams of Acetrol per liter (a commercial inhibitor manufactured by E. F. Houghton and Company), Fig. 14. The potential readings (Table 2) were taken on a recording Brown

Potentiometer Pyrometer, Model No. 151321. These data were all recorded as degrees Fahrenheit but were converted over to millivolts. In order to make a more accurate conversion the Brown instrument was calibrated with a Thwing Potentiometer Checking and Calibration Set which is capable of taking readings down to 0.1 millivolt. After calibrating, the subdivisions on the Brown Potentiometer were equivalent to about 0.4 millivolt. It might be concluded readings within 0.5 millivolt were obtained. With the foregoing arrangement it was possible to obtain continuous potential readings with very little or no current flow.

One difficulty found in this set up was that the limit of the Brown instrument was about 44 millivolts. In starting, some potentials went well above this value. To extend the range of the recording instrument an external potential of known value was placed in series with the Brown instrument, Plates II and III.

In order to check results at least two samples of a given heat treatment were run. To get a visual picture of the agreeance on these tests they were plotted and are shown in Figs. 4 - 12.

DISCUSSION OF RESULTS

It was impractical to express the results as an overall picture because of the overlapping implications. Thus, it was a much better plan to discuss each group of data separately. Since hardness was the only physical property that was actually obtained, this will be discussed first.

Hardness

It can be noted on Plate I and Tables 5 and 4 on the quenched sample, the hardness was high (R_c 65). When tempered at 600° F. to 800° F., it dropped to R_c 61. On tempering at 1035° F. the hardness increased to R_c 64.5.

A very common explanation of the drop in hardness is the tempering of martensite, and the increase in hardness that is obtained is due to the transformation of austenite to martensite.

It has been expressed by some authorities (5, p. 528) (8, p. 48) that carbide forming elements such as chromium, tungsten and vanadium, increase the stability of martensite and austenite, making it possible to have martensite at a temperature of 1000° F. and austenite at room temperature. In any respect the change in hardness produced on tempering is probably due to a changing balance between the amount of austenite and the very hard martensite.

Indications of the austenitic content of quenched high speed steel can be seen under the microscope, Plate IV. When a quenched sample of high speed steel is etched the base metal appears white due to the retained austenite. However, when tempered at 1035° F. or annealed it, appears dark due to the formation of martensite or ferrite whichever the case may be.

Magne-Gage

In an attempt to show the changing austenitic content as the steel was tempered at various temperatures a method similar to the one used by Simpkinson and Lavigne (9) was employed. In this method the Aminco-Brenner Magne-Gage was used to determine the magnetic properties, Fig. 15. This instrument was originally used to determine the thickness of coatings on magnetic material.

The Magne-Gage consists essentially of a small permanent magnet 0.08 inch diameter with hemispherical contact end, suspended from a horizontal bar arm. The lever arm is actuated by a berylium-copper spiral spring which is coiled by turning a knurled knob clockwise. A graduated dial indicates the amount of rotation. In this way the relative amount of force that is required to pull the magnet away from the part can be measured. The numbers on the dial are in reverse order so the large numbers indicate the least pressure and consequently

the larger numbers would indicate the most austenite.
(Austenite is not a ferromagnetic material.)

It has not been determined whether this gives a perfectly accurate representation of the amount of austenite, but it does give an indication.

The curve on Plate I indicating the Magne-Gage reading will reveal that very little austenite was transformed on tempering up to 400° F. Then some was transformed from 400° F. to 600° F. and none transformed from 600° F. to 800° F. From 800° F. on up to 1035° F. there was a very rapid transformation of austenite. In the region of 1200° F. to 1400° F. there was very little austenite transformed.

The Magne-Gage readings compared very favorably with the theories of secondary hardness presented in the foregoing section entitled hardness.

Weight Loss and Potential Difference

In all cases the annealed samples were negative (anodic) to the treated samples and thus connected to the negative side of the recorder. It might also be noted that in all cases the cathode lost more weight than the annealed sample. At first glance this might be contrary to all rules of electrochemistry. It should be remembered that alloys are heterogeneous (made up of more than one constituent). This being true the greater

weight loss on the cathode could be due to a local cell set up in the heat treated parts indicating that the treated samples were more heterogeneous than the annealed samples.

This weight loss was expressed in grams per square inch per hour per 0.001 gram of loss on the anode, Tables 3 and 4. The reason for this means of expression was the fact that the corrosion rate and the potential can be affected by temperature, oxygen content of the electrolyte and various other factors, all of which were very hard to control. However, all of these conditions were the same for the anode and the cathode in a given test.

In the set up used it should be remembered that all circuits were balanced; no current was flowing, thus no weight loss should be due to the galvanic action. If this had been a closed circuit more weight loss would have been expected on the anode or to express it in another way less loss on the cathode. To check this, samples 102 and A2 were re-ground and put in the electrolyte, but instead of putting these in series with the recorder the two electrodes were electrically connected. It was found that the equivalent weight loss was only 0.0148 gram per square inch per hour while the average for samples 10 was 0.0415 gram per square inch per hour, Table 4. This demonstrates that an anodic material in series with a cathodic material will tend to

protect the cathodic material.

Another means of studying the potential would be to take the average for all of the potential readings of a given set of samples, instead of the potential at which they tended to level off, Table 5. Although the curves tend to agree, their averages were not plotted because it was thought that the rough surface condition on the sample might affect the beginning potential. It might be added that a curve of these data agreed with the general shape of the hardness curve.

Table 2. Data for potential time curve.

Sam-		Poten	tial in	m1311v	olta at	the end	ofea	ch hour.	
pres	: 1					: 6 :	7	8:	9
Aı	30.6	30.2	28.8	24.8	20.0	16.8	15.6	15.6	16.5
Q1	16.6	16.5	16.6	16.4	16.4				
A2	28.0	28.0	27.0	26.2	23.0	20.2	17.2	15.6	14.0
Q2	14.6	15.0	15.0	15.2	15.2				
A3	29.8	30.2	27.6	20.0	16.4	16.0	16.4	16.6	16.6
21	16.6	16.6	16.6						
AA	30.6	31.6	29.0	26.0	23.2	21.6	20.2	19.8	17.4
25	17.4	17.4	17.6						
A5	28.4	29.4	30.2	29.2	27.0	23.9	21.2	18.0	15.0
23	15.0	15.0	15.0						
A ₂	23.8	23.8	25.0	23.2	22.0	20.0	20.0	19.5	18.8
42	17.6	17.4	17.4	18.0	18.4	21.5	21.5	21.5	
A ₃	24.5	24.5	23.0	21.0	21.0	19.8	20.2	19.3	19.8
44	20.5	20.5							
Ah	9.0	6.8	8.5	11.8	14.2	16.5	18.0	21.2	22.1
61	25.0	28.0	26.0			2009	2010		
As	11.8	9.0	10.4	11.8	15.0	18.8	19.2	23.2	25.0
62	27.5	30.0	30.0						
A6	25.0	25.0	24.0	23.5	23.5	23.5	23.8	24.4	25.5
63	25.5	24.2							
Al	9.5	7.5	7.7	9.0	11.3	13.8	16.0	17.2	17.2
81	22.2	19.2							
A2	27.5	17.5	13.2	13.2	14.0	15.0	16.2	17.2	18.4
82	19.2	18.9							
A3	14.0	13.5	10.5	13.2	14.1	17.2	18.8	20.2	21.6
83	22.6	24.0							
A ₁	22.8	23.6	24.0	23.6	24.0	25.3	25.6	26.0	26.7
101	26.7	26.7	27.5	29.2					

Table 2 (concl.).

Sam-		Poten	tial in	millivo	olts at	the en	d of ea	ch hour	
	: 10	2 11	3 12	13	5 14	6 15	7 16	8 17	9
A ₂	21.5	23.2	24.5	24.5	25.3	26.5	27.5	28.8	28.8
102	29.4	30.1	30.1	30.1					
A3	23.2	24.0	25.3	25.3	25.0	25.0	25.1	26.0	26.3
103	26.3	25.6	26.3	26.0					
A4	23.6	25.2	25.2	23.6	21.2	19.8	18.4	17.0	20.5
121	12.0	12.0							
As	23.4	24.3	24.3	21.2	18.0	15.5	13.6	11.3	11.0
122	10.5	10.5	11.0						
A6	25.0	25.5	25.0	21.2	19.6	16.3	14.8	12.8	10.0
123	12.2	13.0							
AA	6.8	7.2	7.7	7.2	6.4	5.1	4.8	4.8	4.4
141	4.0	4.5	4.5						
A6	4.3	4.4	5.4	6.2	6.7	6.7	6.7	6.7	6.7
143	6.7								
A ₁	4.0	4.1	4.0	4.4	4.5	4.6	4.8	4.8	4.3
A ₂	4.4	3.6	2.8						
A3	2.6	2.9	2.9	2.9	2.9	2.9	3.4	3.8	4.2
AA	4.2	4.2	4.3	4.0	4.4				

Table 3. Pertinent data obtained from samples tested.

		:loss g.				Oli Bambio	E . TUERE
Al	B97	0.0242	1.640	14	0.00105		27
91	065	0.7625	1.720	14	0.0291	0.0277	57-58
A2	B97	o comp	1.710	14			27
€5	665	0.6978	1.685	14	0.0294		55-57
A3	B97	0.0293	1.666	122	0.00141		27
21	065	0.9446	1.666	128	0.0454	0.0322	58-60
A4	B97	0.0208	1.615	12章	0.00100		
22	065	0.6746	1.482	125	0.0364	0.0364	57-59
A ₅	B97	0.0096	1.538	104	0.00058		
23	c65	0.3459	1.482	103	0.0216	0.0382	56-59
A ₂	B97	0.0125	1.650	178	0.000432		27
42	c63.5	0.9546	1.570	17亩	0.0347	0.0803	57-58
A3	B97	0.0056	1.650	112	0.000295		27
43	063.5	0.4396	1.570	111	0.0243	0.0818	54-57
AA	B97	0.0065	1.475	114	0.000374		27
61	C61	0.4945	1.475	112	0.0285	0.0762	52-49
As	B97	0.0047	1.475	112	0.000270		27
62	C61	0.4098	1.535	112	0.0226	0.0837	52-49
A6	B97	0.0053	1.475	112	0.000312		261
63	C61	0.3112	1.475	112	0.0183	0.0587	51-49
Aı	B97	0.0037	1.491	112	0.000215		
81	C61	0.3758	1.752	112	0.986	0.0865	50-51
A ₂	B97	0.0051	1.775	11章	0.000250		
82	061.5	0.2802	1.455	111	0.0167	0.0669	49-51
A3	B97	0.0037	1.452	11	0.000231		
83	C61.5	0.3916	1.390	11	0.0296	0.1410	50-51
A ₁	B97	0.0085	1.184	134	0.000540		
101	C64.5	0.2431	1.142	134	0.0160	0.0296	36-37
A ₂	B97	0.0053	1.184	134	0.000337		
.02	C64.5	0.2756	1.147	134	0.0181	0.0540	36-36

Table 3 (concl.).

		: Total wt.	Area	time :	: Nt. loss : :6./sq. in.: :/hr. :	g./.001 g.	
A3	B97	0.0064	1.184	132	0.000407		
103	064.5	0.2521	1.420	134	0.0167	0.0410	36-37
AA	B97	0.0122	1.425	112	0.000760		
121	C59	0.2352	1.493	111	0.0140	0.0184	32-33
A ₅	B97	0.0146	1.425	111	0.000885		
122	c59	0.2826	1.555	111	0.0157	0.0176	32-33
A6	B97	0.0161	1.425	11	0.00103		
123	C59	0.2645	1.490	11	0.0162	0.0159	32
AA	B97	0.0076	1.400	12	0.00452		
141	C38.5	0.0094	1.480	12	0.000527	0.00117	27
A6	B97	0.0089	1.400	10	0.000635		
143	038.5	0.0126	1.480	10	0.000890	0.00140	27
Aı	B97	0.0082	1.214	12	0.000564		27
A ₂	897	0.0133	1.214	12	0.000915		27
A3	B97	0.0128	1.214	14亩	0.000725		27
A4	B97	0.0105	1.214	14世	0.000595		27

Table 4. Summary of data in Tables 2 and 3.

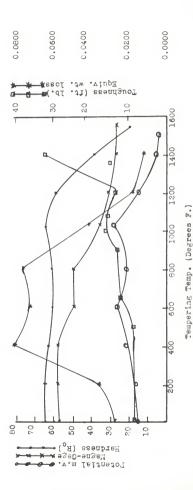
	Rockwell hardness	: Equiv. wt. loss : g./sq. in./hr. : /.001 g. loss : on sample A.	: Magne- : Gage	: : Potential at level- : ing off point
2 4	065 065 063.5	0.0277 0.0359 0.0810	57 58 57	15.9 16.9 22.0
6	C61 C61.5	0.0729	51 51	27.0
10	C64.5	0.0415	36 32	28.4 15.0
14 A	c38.5 B97	0.0128	27	5.6

Table 5. Average potential difference for all tests run.

Sample	; Average potential m.v.
0	18.8
2	18.8 22.6
4	21.3
6	20.3
8	16.1
10	28.1
12	18.1
14	4.9
A	3.0

EXPLANATION OF PLATE I

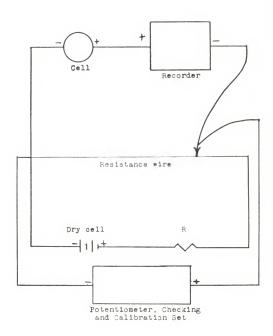
A graphical representation of the data presented in Tables 3 and 4.



EXPLANATION OF PLATE II

This shows the detailed wiring diagram of the set up used in obtaining the experimental data. A photograph of the equipment is shown in Plate III. The limit of the recording potentiometer was about 44 millivolts. To extend the limits of the recorder an external potential was put in series with the galvanic cell and the recorder. The potential produced by the cell was then equal to the potential indicated on the recorder plus the potential indicated on the Potentiometer Checking and Calibration Set.

PLATE II



EXPLANATION OF PLATE III

Arrangement of the equipment used.

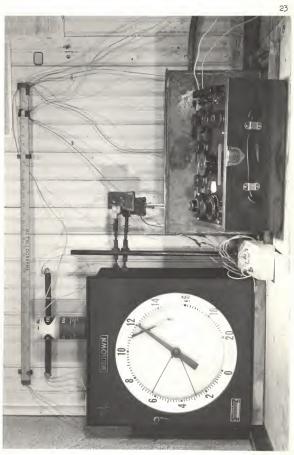


PLATE III

EXPLANATION OF PLATE IV

- Fig. 1. Photomicrograph of a sample quenched at 2350° F.

 Etched in 4 per cent nital for 60 seconds.

 Magnification 530 diameters. Hardness Rc 65.0.
- Fig. 2. Sample quenched from 2350° F. and tempered for one hour at 1035° F. Etched in 4 per cent nital for 60 seconds. Magnification 530 diameters. Hardness $R_{\rm C}$ 64.5.
- Fig. 3. Annealed sample. Etched in 4 per cent nital for \$60\$ seconds. Magnification 530 diameters. Hardness $$R_{\rm b}$$ 97.0.

PIATE IV



F1g. 1



Fig. 2



Fig. 3

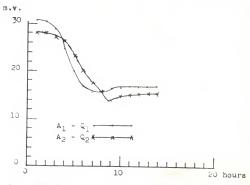


Fig. 4. Quenched sample positive and annealed sample negative. Potential vs. time curve.

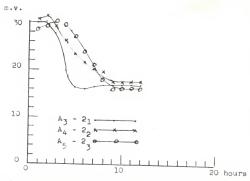


Fig. 5. Sample tempered at 200°F. positive and annealed sample negative. Potential vs. time curve.

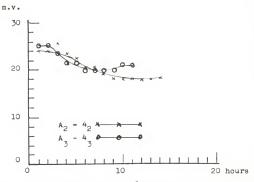


Fig. 6. Sample tempered at 400° F. positive and annealed sample negative. Potential vs. time curve.

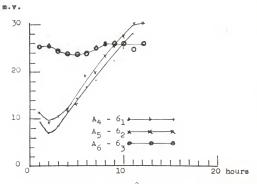


Fig. 7. Sample tempered at 600°F. positive and annealed sample negative. Potential vs. time curve.

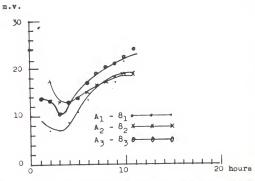


Fig. 8. Sample tempered at 800°F. positive and annealed sample negative. Potential vs. time curve.

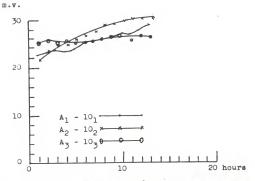


Fig. 9. Sample tempered at 1035°F. positive and annealed sample negative. Potential vs. time curve.

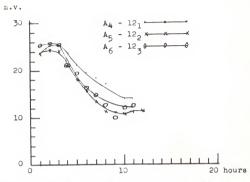


Fig. 10. Sample tempered at 1200°F. positive and annealed sample negative. Potential vs. time curve.

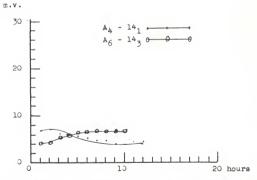


Fig. 11. Sample tempered at 1400°F. positive and annealed sample negative. Potential vs. time curve.

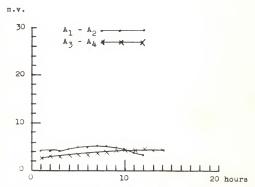


Fig. 12. Annealed sample vs. annealed sample. Potential vs. time curve.

EXPLANATION OF PLATE V

- Depicting the samples, the method of making electrical contact, and details of masking. F16. 15.
- containers, electrolyte and the arga-arga KCL bridge. Showing details of Galvanic Cell, including samples, Fig. 14.

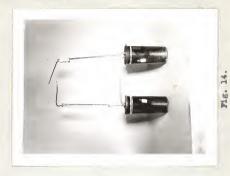


PLATE V

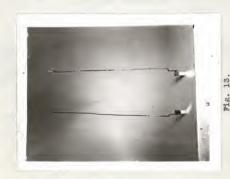




Fig. 15. A front view of the Aminco-Brenner Magne-Gage.

CONCLUSIONS AND SUGGESTIONS FOR FURTHER STUDY

When the material was tempered at progressively higher temperatures up to 1035° F., the positive potential increased, at this temperature the maximum usable hardness was obtained. The increase in potential could be due to a balance between the following factors: 1. carbides being precipitated out from martensite which would tend to decrease the hardness, 2. austenite changing to martensite which would tend to increase the hardness, 3. the relieving of internal stresses set up in the material.

Tempering at temperatures above 1035° F., the potential difference decreases very rapidly due to the fact that the structure of the two electrodes were becoming more alike. This was also indicated by the leveling off of the Magne-Gage readings.

As stated in the discussion the weight loss was due to the heterogeneous nature of the material and there was a very close correlation between potential and Magne-Gage readings. It might be concluded that the Magne-Gage reading would be a measure of heterogenecy.

It might be stated that due to the complexity of the material definite conclusions could not be drawn, only generalizations.

This work has brought out some definite points: 1. There

is a difference in potential for metals tempered at different temperatures. 2. This difference can be measured and results duplicated. 3. This work brought out the possibility of using the Magne-Gage as a means of measuring the amount of austenite in high speed steel.

The method might be a very fruitful field for further work. To get away from a complex steel a plain carbon steel of sutsectoid composition should be run by using the same method as described in the work. It might be helpful if a method of electrolytic polishing could be worked out to obtain a better surface condition on the samples.

Another research problem would be to standardize the Magne-dage as a method of determining austenite in steel.

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