















EFFECTS OF NABCENT HYDROGEN ON IRON AND STEEL AT ORDINARY TEMPERATURES

A thesis presented to the Academic Faculty of the University of Virginia in candidacy for the degree of Master of Arts.



#### EFFECTS OF NASCINT HYDROGEN ON IRON AND STEEL

AT ORDINARY TEMPERATURES

PART I - PRELIMINARY REPORT.

Among the first experiments to determine the effects of hydrogen on the physical properties of iron and steel were those of Coulson in his efforts to determine some method of pickling steel, intended for use in spring construction, without the attending brittleness usually found in the product. He used for the pickling solution a bath of 27% H<sub>2</sub>SO<sub>4</sub> at60°C. He found that specimens of spring steel pickled 40 seconds chemically or as cathode, when tested for endurance, broke during the preliminary tests, while the untreated specimens and those treated as anode showed a life of about 10,000,000 vibrations before breaking.

In bending tests, pieces which had been pickled chemically and as cathode electrolytically, showed fatigue and cracks much sooner than untreated test pieces or those which had been treated as anode. Spring steel wire untreated or treated as anode broke at 253,000 lbs/si and reduced only51% in area. Drill rod and cold rolled steel specimens showed differences in breaking stress and percentages of reductionin area, but not so marked as in spring steel wire mentioned above, which broke at 247,000 lbs/si and with a reduction of only3% in area after treatment chemically or as cathode in the solution. The properties of high carbon steel were seen to undergo the greatest modification.

The same writer states that test specimens, made brittle by pickling either as cathode or chemically, were



restored to the original condition by annealing. This restoration was effected to a considerable degree by repickling the pieces as anode. The writer concludes that it is probable that prolonged repickling electrolytically would restore completely the physical properties of the metal.

T.S.Fuller, in the laboratories of the General Electric Company, found that immersion in a 1% solution of H<sub>2</sub>SO<sub>4</sub> gave a greater penetration of hydrogen than when the test pieces were treated electrolytically as cathode in the same solution. He also found that coating the specimens with tin increased the rate of penetration of the hydrogen while coating with copper produced the opposite effect. Dipping the specimens in molten copper was found very effective in preventing the absorption of hydrogen.

Earle A.Harding and Donald P.Smith made a series of tests to determine the effect of occluded hydrogen on various metals. While most of their experiments were made with palladium, still the results of a few tests on iron and steel led them to believe that the same results would hold for these metals which were found to be true for palladium. The main things determined were the electrical resistance and physical expansion of the specimens under treatment. The resistance of the pieces was noticed to decrease with the absorption of the first increments of the hydrogen, supposedly due to the addition of current carrying free hydrogen. However, as the process went on, the resistance was noticed to increase gradually, due perhaps to the combination of the hydrogen with the metals to form various

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non-conducting alloys. At the same time the linear dimensions of the test pieces were noticed to increase; the rate being greater, proportionally, for the last increments of hydrogen absorbed. This fact indicated that the expansion of the metal was due to the pressure of the occluded hydrogen.

Fuller describes in the G.E.Review another set of experiments with a specially devised apparatus for determining the effect of variation of temperature, electric current, electrolyte and previous treatment of the specimens and surface coating of the test pieces on the specimens by nascent hydrogen. His results, briefly summed up, are as follows:

An increase of the current caused an increase of the rate of penetration of hydrogen, not in proportion, however, to the increase of current.

Heat increased the rate of penetration, the same amount of gas penetrating at  $90^{\circ}$ ,  $80^{\circ}$  and  $20^{\circ}$  in 3.5, 4.75 and 48 hours respectively.

A 1% solution of sulphuric acid proved a better electrolyte than  $K_2SO_4$ , KOH or tap water. However, steam, by its action on iron, produced a greater penetration of the hydrogen than was caused by electrolytic treatment in the acid solution.

Charpy and Bonnerot found iron impermeable to gaseous  $(H_2)$  at temperatures as high as  $325^{\circ}$ C. Still the iron became increasingly permeable from that temperature on. Pressures as high as 26 atmospheres had no effect in forcing permeability. The same investigators observed that iron

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acts as a kind of membrane permeable to nascent hydrogen. This was observed by allowing hydrogen to be liberated from the attack of the acid on the iron membrane; also when the specimen was made the cathode in an acid solution Osmosis occurred only when hydrogen was liberated directly on the membrane. By means of a very interesting piece of apparatum it was found that pressures as high as 14 atmospheres were produced in a closed chamber, due to the osmotic pressure exerted by the hydrogen.

Andrew decided, from experiments on iron with caustic soda, that hydrogen, upon being occluded by iron, immediately gave rise to large crystals and consequent brittleness. He attributes the crystallization to a molecular disturbance caused by the occluded hydrogen.

Stromeyer observed the effect of cold caustic soda on mild steel. He found plates and tubes to crack and rivet heads to fly off. However, this brittleness and corrosion occurred onlywhen the specimens were in tension. No effect was observed when the metal was unstressed or in compression.

Quite a number of investigations have been conducted from time to time to determine the effects of hydrogen at ordinary temperatures on iron and steel. Then there has been a vast amount of research in the same line at higher temperatures. Since the present study is confined to the effects at ordinary room temperatures, only a few of the representative results that have appeared from

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research in this particular branch of the subject have been mentioned briefly.

An article by John Goulson, appearing in the Physical Review of July 1922, should be given a little fuller treatment here. The subject of his research was " The Effect of Nascent Hydrogen on Hard Steel Magnets." The following quotation from the article mentioned above describes the author's apparatus and methods used in his research.

"The measurements were made by placing on the opposite side of a magnetometer a standard bar magnet in Gauss's A position, held, and protected completely from rapid temperature changes, within a wooden carriage sliding on a horizontal scale. This compensating magnet, having been fixed with its center at a convenient distance,  $d_0$ , from the center of the magnetometer needle, brought the needle back into the meridian when the center of the magnet under observation was at the fixed distance, d. Let  $M_0$  be the magnetic moment of the standard magnet and  $\mathfrak{M}_0$  its magnetic length, and let  $M_1$  and  $M_1$ ' be the moments of the magnet under observation, before and after treatment respectively, and let 2L be its magnetic length. The needle will be deflected thru the angle  $\alpha$  so that

$$\frac{2M_{o}d_{o}}{(d_{o}^{2}-l_{o}^{2})}=\frac{2M_{i}d}{(d_{o}^{2}-l_{o}^{2})^{2}}$$

 $\frac{2M,d}{(d^2-l^2)^2} - \frac{2M,^*d}{(d^2-l^2)^2} = \frac{2d(M,-M,)}{(d^2-l^2)^2} = H \tan \alpha,$ 

$$\frac{M_{i}-M_{i}}{M_{i}}=\frac{(d_{o}-l_{o})}{ZM_{o}d_{o}}, H \tan \alpha = \frac{\tan \alpha}{\tan \alpha_{o}}.$$

where H is the horizontal component of the earth's field, and  $\alpha_o$  is the angle through which M<sub>o</sub> would deflect the needle if M<sub>o</sub> were absent.

"The method of procedure was then as follows: "The standard magnet was placed with its center at a known distance from the magnetometer, and its



"carriage clamped firmly in position. The magnet to be tested was placed on the opposite side of the magnetometer and adjusted in such position as to bring the needle back into the meridian. Its holder was then clamped and the distance of the center of the magnet from the magnetometer was recorded. After subjecting the magnet to treatment, it was again placed in its holder and the deflection of the magnetometer needle observed with the magnet at room temperature. The dimensions and magnetic moment of the standard magnet being known, the magnetic moment of the test magnet and fractional loss of moment could easily be calculated."



#### EFFECTS OF MASCENT HYDROGEN ON IRON AND STEEL AT ORDINARY TEMPERATURES

PART II - CANDIDATE'S INVESTIGATIONS

#### Tests on Magnets

The article from which the quotation was taken suggested the work undertaken by the writer and used as the subject of this thesis. The investigations were carried on with materials at ordinary room temperatures. The solution used was 25% sulphuric acid, and the current who maintained at two amperes during treatment.

The first work done was an attempt to check the results of Coulson's experiments on magnets. The apparatus used was not designed to give actual readings of the moment of the magnets tested, nor to give data from which the moment could be calculated, but rather the relative moment before and after periods of treatment.

The magnetometer and carriage for the magnet under test were arranged as described in the Coulson experiments. However, in place of the movable standard magnet, the balancing effect was produced by a fixed coil through which a current from the storage battery could be passed. The magnitude of the current was regulated by means of slide wire rheostats. Any change in the magnetic moment of the test magnets was readily detected by the change in the current necessary to restore the needle of the magnetometer to the neutral position. Throughout a series of tests on a specimen, the carriage holding the test piece as well as the balancing coil remained firmly clamped to the supporting bar.

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Magnets were prepared from tool steel by first hardening the steel by quenching in cold water from red heat. Then the specimens were magnetized in the field of the laboratory electomagnet.

Table # I gives the results of tests on a series of magnets prepared in this way. The preliminary ageing was accomplished by repeatedly heating the specimens to the temperature of boiling water and cooling. The magnets were placed in test tubes and then immersed in the boiling water. Still some moisture got into the tubes in nearly every instance. The fact that a portion of the lost moment of the magnets appeared to be regained when the specimens were treated as anodes, suggests the possibility of a slight occlusion of hydrogen from contacy with water vapor during the heating process.

A new series of magnets were made the cathode in the acid solution. The moment is seen from the table to drop an amount sufficient to balance a current in the coil ranging from 1 to 8 malliamperes for the various specimens. When magnet #5 was treated as anode for 30 minutes its original moment was restored.

On first consideration, due to the smallness of the change in moment due to the hydrogen treatment, it was thought to be the result of some defect in the apparatus or else some error in the manipulation. However, on closer study of the results, they seem to be compatible with what might be expected.



The apparatus used in treating the specimens with hydrogen consisted of a lead lined glass jar of acid placed on an adjustable clamp stand, to which a second clamp was attached above the jar and held an electrode. This electrode when in approximate contact with the magnet standing upright in the middle of the jar, could be screwed down firmly against the end of the specimen, at the same time holding the magnet in position and also furnishing a good electrical contact. The lead lining furnished the second electrode. The rheostats for regulating the current were placed in the vertical position in order to eliminate the possibility of a disturbing electrical field in the vicinity of the magnetometer. The photograph below shows very clearly the arrangement of apparatus.



Apparatus used in Experiments on Magnets (Photo by Weed)

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#### Tests for Moduli of Rigidity and Elasticity

The next experiments undertaken were for the purpose of determining the effects of hydrogen treatment on the modulus of rigidity of several specimens of hand piano wire. The first tests were made on 18" specimens cut from a roll of hard piano wire of about 0.03" diameter. This particular length was chosen in order to have the pieces of proper length for use in the wire testing machine located in the Engineering laboratory. Since the test pieces could not be easily straightened it was necessary to coil them more closely and hold them in a stressed condition during treatment. In nearly every instance the specimen either cracked or broke completely. An examination of the specimens showed a very marked brittleness after treatment while the untreated wire could be kinked without breaking.

Specimens were then taken from a stock of specially prepared piano wire of 0.045" diameter. The pieces were cut in 18" lengths and were perfectly straight, thus making excellent stock for test purposes. A deep jar was prepared with lead lining as was the first. The depth of this jar allowed the pieces to be treated without bending. After treatment it was noticed that the specimens could be bent into kinks almost as readily as before treatment. It was thought that this difference in brittleness of the two winds of wire was possibly due to a difference in composition of the two metals. So specimens from other wire were chosen for further investigation.

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Later the report of Stromeyer was found, in which he attributes brittleness in iron containing occluded hydrogen to the stressed condition of the iron during treatment. Then the second kind of piano wire which showed no brittleness resulting from the first treatment, was held in a coiled condition while hydrogen was applied. It also broke during treatment. The brittleness of the specimens treated this way proved the theory of Stromeyer very conclusively.

A third specimen from 1/8" stock of soft iron wire was chosen and treated in the natural unstressed condition. it was found after treatment that one reversal of a right angled bend was sufficient to fracture the piece, while before treatment four reversals were uniformly required to break the wire. This shows that in some cases at least a slight brittleness is produced even in iron treated in an unstressed condition. However, the effect is not nearly so great as that produced in specimens treated under tension.

The specially prepared piano wire mentioned above, of 0.045" diameter was tested by the torsion pendulum method for change in rigidity modulus due to mascent hydrogen. Table II shows the results of this series of experiments. The modulus of rigidity is seen to be materially decreased in iron thoroughly permeated by the hydrogen. Numbers of specimens, both untreated and treated were suspended from a clamp and supported as a bob a small brass cylinder<sup>\*</sup> This bob when set in vibration was timed in each case

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for 100 complete oscillations. By means of a 1/4 second states by watch, readings of a remarkable accuracy were obtained as is seen by the repeated checks in the readings.

The same specimens were used in the wire testing machine for a determination of the elastic limits. However, the machine was too heavy for wire of such small cross section and the readings obtained were of little or no value on that account. Table III and Plate II show the data and curves made from the results of this experiment. The curves show a tendancy opposite to that which would naturally be expected from the tests on brittleness.

The most satisfactory results of all were those obtained in the tests for change of modulus of elasticity due to hydrogen. Two specimens from a 3/8" rod of cold rolled steel were prepared with threads at the ends for use in the Modulus of Elasticity Machine in the Engineering laboratory. One of the specimens was treated as cathode in the acid for two hours. The lead container served as anode in this test as a special arrangement had to be used in this test. The second specimen was left untreated. Measurements showed no decrease in diameter due to the acid bath for two hours.

The results of the test on the two pieces are shown in Table IV. The curves and coefficients of Young's modulus derived from the data appear on Plate III. A glance at the curves show at once the change that his resulted from the acid and hydrogen treatment. It

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appears that the molecular structure of the steel has undergone some change sufficient to materially modify the elastic properties of the metal.

# Trsts for Elongation due to Hydrogen

During the experiments on magnets an attempt was made to determine whether the hydrogen treatment produced any change in the linear dimensions of the magnets. The dividing engine was used in this attempt. The marks on the specimens could not be maintained sharply enough defined to give results of any value.

During the tests on the piano wire the attempt was again made to determine whether the wire lengthened any due to the occluded hydrogen. This time a different method was tried. Two sliding clamps were fatted on a steel rod. The clamps were spaced just far enough apart to admit the untreated specimen of wire. Then after t treatment the specimen was again traded in the clamps to determine any change in the length which might flave occurreed during treatment. No change was noticed in the several specimens tried in this way.

#### Tests of Thermo-Electric Effects

Experiments were und rtaken to determine what change treatment with hydrogen made on the thermo\_electric properties of iron and steel. The aparatus first used was a thermos bottle containing crushed ice to keep the cold junction at zero, while the hot junction was maintained at steam heat by means of a hypsometer. Two methls used were copper and steel. The couple was first with the



steel untreated, after which the hydrogen treatment was applied and the experiment repeated. This method failed to produce results of value and was discarded for the hot-cold plate method, in which a change of homogeneity of the material of a wire may be detected between very closely adjacent points.

For a hot plate the bottom of a hypsometer was used. This plate was kept hot by running live steam through it from a second hypsometer. Through the vessel serving as cold plate a constant stream of tap water was passed. The two plates were separated only about one fourth off an inch. The plates were covered with shellacked paper for electrical insulation from the test wires.

Two wires of the same material as that under test were attached directly to the terminals of a sensitive galvanometer. These were used as exploring wires, the free endsbeing in contact with the wire under test at points about two centimeters apart. One contact point was in contact with the hot plate and the other with the cold. The test wire was then moved two centimeters at a time over the plates , the exploring wires being kept stationary. At each new setting the deflection of the galvanometer was read and used as an indication of the E. . . of the couple formed by the exploring wires and that particular portion of the test wire between them. The tests were repeated under as nearly identical conditions as could be produced, in order to test the accuracy of the method. Such widely varying results were

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obtained that an effort was made to increase the accuracy by soldering copper leads to the exploring wires. These leads could then be clamped more firmly to the galvanometer terminals.

The tests were then repeated on a new specimen. As before, the readings were not found to check the corresponding readings intwo trials. Still the specimen was treated by hydrogen to a distance of about six centimeters from one end and the tests were repeated twice more. Again readings which resembled very closely those taken in the tests on the untreated specimen were made. No change occurred to indicate a difference due to the hydrogen treatment.

Table V gives the recorded readings obtained in these tests. Plate IV shows a diagram of the electrical circuit.

# Tests for Change in Crystalline Structure

The last series of tests were made to determine any change in the texture of specimens of cold rolled strip steel after treatment with nascent hydrogen.

A microscope of as high magnifying power as was feasible to use was tried in these tests. A higher power objective lens was tried but sufficient light could not be supplied to the material under observation when it was in such close proximity to the objective lens.

The first specimen was polished carefully and treated as cathode in the acid solution for about an hour. It was then removed and polished a second time. A second



Diece from the same strip was also polished and the two etched by means of nitric acid. It was noticed that the degree of etching made a difference in the appearance of the metal under the microscope. Still no difference in t the crystalline structure of the two pieces could be detected.

The experiment was repeated on two new specimens from another strip of steel. The treated piece this time was held in tension during treatment. Still no visible difference in texture was noticed.

Magnets, broken after treatment, when compared with broken sections of untreated magnets seemed to show under slight magnification a difference in appearance for the portions penetrated and that not entered by the hydrogen. However, the difference was not marked enough to check the statements of Coulson reparding his tests on the same thing. He says a different appearance results because of a change of the crystal structure due to the hydrogen. The break occurring along the crystal faces allows the light to be reflected from these faces and consequently a brighter apparance of this portion of the metal shows the depth of penetration of the hydrogen. Photograps of broken sections seem to prove his theory.

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# TABLE II

RESULTS OF MODULUS OF RIGIDITY TESTS - TORSION PENDULIM METHOD

Modulus of Rigidity  $= n = 8\pi lI/t^2r^4$ I = moment of inertia of bob l = length and r\_ radius of the test wire t = period of the pendulum

Spe	ciaen	Trials	Period of Treatment minutes	Period of Pendulum seconds	Time of 100 Vibration:	3	n
	1	Av of 3	2	1.53	153	361	x10 <sup>6</sup>
	2	5	4	1.52	152	365	11
	3	3	6	1.52	152	365	H
	4	3	8	1.54	154	356	n-
	5	3	10	1.54	154	<b>35</b> 6	8
	6	3	15	1.54	154	3 <b>55</b>	11
	7	3	20	1.52	152	365	11
	8	3	30	1.52	152	365	11
Unt	reateds	pecimens					
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	10	3	0	1.55	155	351	ŧŧ
	11	3	0	1.60	160	330	83
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3	0	1.5325	153.25		
1	30	1.545	154.5		
2	30	1.5425	154.25	356	. 11
3	30	1.5425	154.25		
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# ROUSS PHYSICAL LABORATORY-UNIVERSITY OF VIRGINIA

DATE

NAME Straley

PARTNERS' NAME

EXPERIMENT NO

TITLE Eff. (H) on Iron & Steel

Tensile Steength of Piano Wire

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TABLE III

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ROUSS PHYSICAL LABORATORY UNIVERSITY OF VIRGINIA

DATE

NAME Straley

TITLE Eff. (H) on Iron & Stell

EXPERIMENT NO

Tensile Strength of Pinno Wire

Diameter of Wire 0.045" Area of Section 9.0016 st APPROVED BY

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TABLE III

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ROUSS PHYSICAL LABORATORY-UNIVERSITY OF VIRGINIA

DATE

NAME Straley

Straley PARTNERS' NAME

EXPERIMENT NO

TITLE Effects of (H) on Steel

APPROVED BY

Test For Change In Modulus of Elasticity Due To Occluded (H)

Diameter of Specimens 0.375" Area " 0.1105 si

			1F	200			#2	
1			Untrea	ted Ro	bd	(H) Tr	eated 2	hrs
	Lood	Strong	Exten-	Extens	Strain	Ext/10	Ext	Strain
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20	400	1500	15 3	106	1325	15.5	140	1324
25	500	4520	16.5	130	1625	17.0	170	1625
30	.600	5420	19	160	2000	18.5	204	2000
35	700	0000	10 5	190	2375	20.2	230	2374
40	800	8145	01	220	2750	21.5	260	2750
45	900	0149		246	3075	23.0	290	3075
50	1000	9050	22.2	076	3450	24.5	316	3450
55	1100	9955	23.8	210	7751	25.8	344	3751
60	1200	10000	25.1	330	2121	27.2	390	4162
65	1300	11765	20.9	200	45.0	29.5	404	4500
70	1400	12670	28.0	200	4075	30.2	430	4875
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80	1600	14480	31.0	420	5250	21.5		2020
85	1700							

TABLE IV

ROUSS PHYSICAL LABORATORY-UNIVERSITY OF VIRGINIA

PARTNERS' NAME

TITLE BITOCOR OF (H) OR BLOG

Y Test For Onange In Modulus of Mantielly Due To Cooluded (H)

Diameter of Speciarns 0.575" Area "..." 0.1105 at

EXPERIMENT NO

NAME STREET

APPROVED BY

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- STEA		9-30-4Q	2373	021 - 18.01-			40
				24. 1 220	Curs. ? .	900	35
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	dit			23-10 276	100055	-100	
1618		Der -	Test.	100 114.24	10050		60
20+m	200E	S. 25 .		- 16×5- 330	11765	1300	- 23
3964	404	5.00	1993A	26. get 1 350	12670	ants	20
4875	430	3012		and ingites		2021	
oct e		34.5	19252	91. D = 490	genna!	0021	

TABLE IV

DO NOT FOLD THIS SHEET

RESULTS THERMO COUPLE TESTS ON HARD PIANO WIRE

Untreated Wire #1

Portion of wire from treated end	E.M.F. Indicated	by Def.of Gal. ans 2nd trial
1st 2 centimeters	1.5	2.5
2nd "	1.5	2.5
3rd "	2.5	3.0
4th "	3.5	1.5
5th "	2.5	2.5
Wire #2 (Untreated.)		
1st 2 centimeters	1.0	1.0
2nd "	0.0	-1.0
3rd "	3.5	1.5
4th "	1.5	2.0
Wire #2 (Treated 6 cen	timeters)	
1st 2 centimeters	3.0	0.5
2nd <sup>11</sup>	2.5	1.5
3rd "	3.5	1.0
4th "	2.5	1.5
5th "	1.5	1.5

TABLE , V





Diagram of electrical connections Thermo-electric tests

PLATE IV











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