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Mossbauer Study of Hydrogen Diffused in Iron and Steels

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The effect of hydrogen on steels has been a perennial object of interest and of research among the makers and users of steels. Hydrogen embrittlement is a problem, especially in the use of high strength steels, for example, in the aircraft and the fastener industries. This investigation is an attempt to use the Mossbauer effect in the study of the effect of hydrogen on iron and steels.

Method

When hydrogen adsorbs on the surface of iron, it quite readily diffuses throughout the piece of iron. The rate of diffusion is proportional to the fraction of the surface of the sample which is covered with hydrogen. It is assumed from the principles of simple diffusion that hydrogen saturation will be reached when the surface coverage fraction is unity.

The method of achieving hydrogen adsorption onto the surface by means of cathodic charging is here preferred over the simpler means of forcing adsorption by high pressures or by sparking for two reasons. More surface area can be covered at room temperature by electrolytic means and greater control of the amount of surface coverage is obtained by the electrical regulation of the current density.

Several experimental obstacles associated with cathodic charging methods had to be overcome before suitable Mossbauer spectra could be obtained. There is the problem of severe attenuation of the 14.2 kev Mossbauer gamma radiation by the steel foil sample itself and by the electrolyte in the path of the radiation. The inhomogeneity of bubbles of escaping hydrogen molecules also present some difficulty.

Experimental

The problem of attenuation by the sample was corrected by using iron and steel foils of thicknesses no larger than 0.001 ". It was found that a path-thickness of about 0.1 " for an aqueous electrolyte balanced the attenuation by the solution and the need for sufficient solution to carry the current. The problem of inhomogeneous bubbles was mollified by the addition of a soap solution to the electrolyte in order to reduce the surface tension of the liquid and by taking many complete scans of the Mossbauer spectrum in the 24 hour period.

Fig. 1 is a diagram of the apparatus used in this preliminary study. A potential difference was supplied by a stabilized power supply (E) and passed across a variable resistance (R). An electrolyte (0.1 N H_2SO_4) was then added to the cell. The resistance R was made large compared to the resistance (r) of the cell, i.e., $R \gg r$, in order to

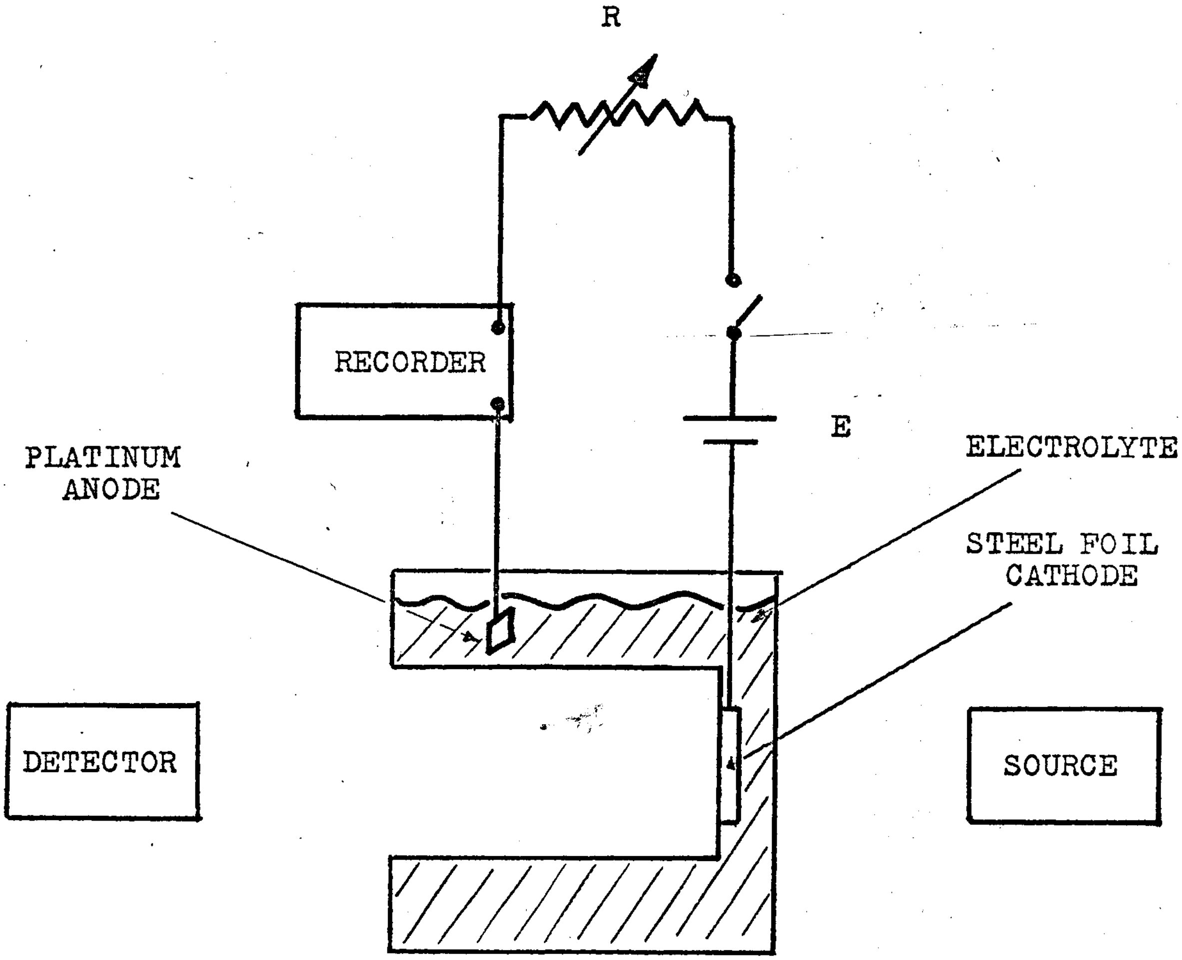

Fig. 1 DIAGRAM OF AN ELECTROLYTIC CELL MOVING ON A CONSTANT VELOCITY MOSSBAUER ANALYZER.

avoid any fluctuations in the current from the electrolysis process. The sample was pre-electrolyzed for 30 minutes in order that the diffusion reach an equilibrium steady-state.

A 6 mC cobalt-in-palladium matrix was used as the Mossbauer gamma radiation source with the steel foil and electolytic cell as the moving absorber. A six line steel spectrum with a baseline of 150,000 counts could be taken in 24 hours using a constant velocity spectrometer with a single channel analyzer. A recorder monitors the constant current of the cell.

Preliminary Results

Three steels of suitable thickness were obtained and submitted to the above procedure. A piece of shim stock steel and a piece of commercially available U.S. steel with a tin coating both exhibiting a well resolved six line spectrum (the ratio of line separation to f.w.h.m. is of the order of 7) readily absorb hydrogen. The permeation and diffusion of hydrogen was verified manometrically and by the observation of blistering on the back side of the cathodically charged foil. A piece of non-magnetic stainless steel 302 having a single Mossbauer absorption line was also subjected to the same process. Hydrogen does not readily diffuse through this steel -- no blistering took place nor was any manometer change observed. Preliminary results of the Mossbauer spectra are given in Table I. Three of the six lines are presented for the magnetic steels.

A general trend of broadening with a corresponding peak-height diminution with the increase of hydrogen production is observed. No conclusions are drawn from this at the present time. The initial observation to be made is the fact that the presence of hydrogen in the lattice does not greatly alter the Mossbauer spectrum. This absence of any large effect will be the subject of further theoretical investigation. This absence, for example, may throw light on the theory that hydrogen atoms ionize in the lattice area during diffusion.

Table I. Preliminary Results of the Mossbauer Spectra

	Current (ma/sq in)	Peak Position* (mm/sec) (1)	(2)	(3)	Line Width (mm/sec) (1)	(2)	(3)	Absorbance (%) (1)	(2)	(3)
Tin-coated Steel	0	-5.47	-3.14	-0.96	0.47	0.35	0.34	10.8	9.3	6.9
	100	-5.51	-3.20	-0.98	0.51	0.37	0.35	10.6	9.5	6.8
	200	-5.51	-3.19	-0.98	0.54	0.39	0.37	10.3	9.3	6.5
	400	-5.52	-3.18	-0.98	0.56	0.42	0.41	9.5	8.6	6.0
Shim Stock Steel	0	-5.51	-3.17	-0.96	0.49	0.37	0.28	10.2	9.9	7.1
	213	-5.49	-3.16	-0.97	0.49	0.39	0.29	10.7	9.8	6.9
	400	-5.51	-3.21	-0.99	0.54	0.40	0.36	10.3	9.2	6.4
Stainless Steel 302	0		-0.27			0.43			22.6	
	20		-0.27			0.44			22.4	
	40		-0.28			0.45			23.3	
	60		-0.29			0.45			23.1	
	80		-0.28			0.46			22.8	
	100		-0.29			0.46			22.7	
	200		-0.28			0.45			22.1	

*Velocities are given relative to cobalt in palladium.