

QUANTITATIVE DETERMINATION OF HYDROGEN IN STEEL

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The electrochemical machining, or chemical etching, of steels in an acid or alkali solution causes a sharp deterioration in their physicomachanical properties as a result of hydrogenation. The degree of hydrogenation is usually assessed by mechanical methods: from the change in the percentage contraction of the cross-sectional area or from the elongation of tensile specimens, by twisting, bending, etc. Direct methods of determination of hydrogen in steel* such as vacuum melting [1-5] or vacuum heating [1, 3, 6] are used less frequently.

During vacuum melting the electrolytic hydrogen content is defined as the difference between the total hydrogen extracted after the electrochemical treatment of the steel and the metallurgical hydrogen determined previously in the initial specimens. However, the method of vacuum melting can be used in determining the electrolytic hydrogen content only if the amounts of the metallurgical hydrogen in various specimens are equal and relatively small. The equipment needed for vacuum melting is very bulky and complex.† For this reason a simpler method of vacuum heating at various temperatures is used for determining the electrolytic hydrogen content: that of heating from 100 to 800°C [7-13]. Various papers fail to suggest a precise procedure for selecting the heating conditions. Some authors suggest that the metallurgical hydrogen should be extracted at temperatures below the melting point (200-800°C) [14, 15] and that it can be expected that during analysis it will be extracted simultaneously with the electrolytic hydrogen. To clarify these points it is interesting to study the effect of temperature on the rate and degree of separation of electrolytic and metallurgical hydrogen from several steels.

The investigation was concerned with Armco-iron and 30KhGSA, U8, and A12 steels. The amount of hydrogen and the duration of extraction depend on the size of the specimens. Cylindrical specimens with a length of 30 mm and diameter of 5 mm were used in all tests. Some specimens were hydrogenated by subjecting them to cathode polarization in a 0.1 N solution of H_2SO_4 for 1 h at a current density of $i_c = 25 \text{ mA/cm}^2$. After electrolysis the specimens were washed in distilled water, acetone, and carbon tetrachloride whereupon they were analyzed. The time from the end of cathode polarization to the beginning of the analysis was 2-3 min.

The amount of hydrogen contained in the steel was determined by the vacuum-melting method and the vacuum-heating method. Melting was carried out in an induction furnace at 1600°C using a GLP-15 tube-type HF generator. The vapor was analyzed on the GEOKhI device [3]. The maximum value of the no-load correction in 20 min was less than 0.15 ml/100 g of metal. The apparatus described in [5] was used in determining the hydrogen content by the vacuum heating method but was modified by the addition of a palladium filter for the separation of hydrogen from other components of the gas mixture.

Best results when degassing steel can be achieved by melting the specimens in a vacuum. The results show that Armco-iron and A12 steel contain from 1.4 to 1.8 cm³ of hydrogen in 100 g of metal while 30KhGSA steel contains 1.1-1.5 and U8 steel 0.7-1.0 cm³ in 100g. The scatter is apparently caused by the nonuniform distribution of metallurgical hydrogen over the length of rods from which the specimens were made.

In determining the metallurgical hydrogen content by vacuum-heating, the specimens were heated to 200, 400, and 600°C. Figure 1 shows the dependence of the amount of gas extracted from 30KhGSA steel for various heating times and at various temperatures. As the temperature increases the rate of extraction and the amount of separated metallurgical hydrogen increase. During the degassing which lasts for over 300 min the rate of extraction reaches a constant value for each temperature. The amount of gas extracted at the established extraction rate

*The hydrogen permeating into the steel during electrochemical machining is referred to as electrolytic hydrogen while the gas contained in the initially untreated specimens will be called here metallurgical hydrogen.

†Some possible errors associated with the use of this method are described in [4].

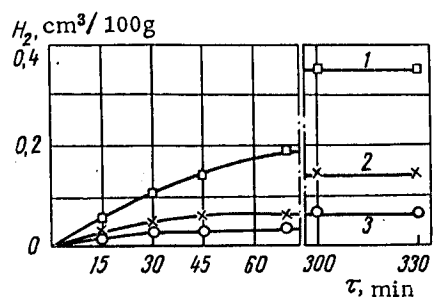


Fig. 1

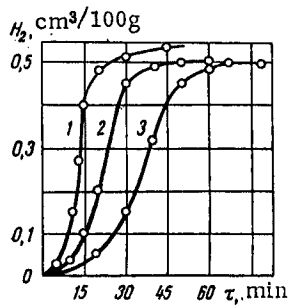


Fig. 2

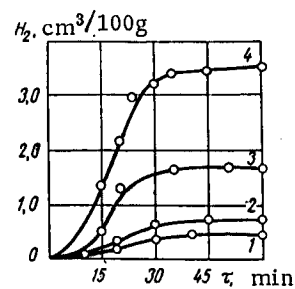


Fig. 3

Fig. 1. Dependence of the amount of metallurgical hydrogen separated from 30KhGSA steel on extraction time: 1-3) extraction temperatures 200, 400, and 600°C, respectively.

Fig. 2. Dependence of the amount of electrolytic hydrogen separated from 30KhGSA steel on the extraction time: 1-3) extraction temperature 200, 400, and 600°C, respectively.

Fig. 3. Dependence of the amount of electrolytic hydrogen separated from various steel grades at 400°C on the extraction time: 1) 30KhGSA; 2) Armco-iron; 3) U8; 4) A12.

TABLE 1. Degassing Time (min) for Various Steel Grades

Temperature, °C	Armco-iron	30KhGSA	U8	A12
200	65-75	70-85	75-90	100-160
400	40-45	40-45	35-40	40-70
600	20-30	20-30	20-35	35-45

depends on temperature. The comparison of results obtained by the vacuum-heating method with those obtained by the vacuum-melting method shows that at temperatures of up to 600°C (this is below the melting point) the metallurgical hydrogen is not completely removed.

Figure 2 shows the extraction of electrolytic hydrogen from 30KhGSA steel at 200, 400, and 600°C. The amounts of hydrogen separated at different temperatures are approximately equal although the desorption time of gas varies. Similar results have been obtained for U8 and A12 steels and Armco-iron. For

each temperature the time needed to remove completely the electrolytic hydrogen from the steel specimens of the above size was measured (Table 1).

The table shows that the degassing time is determined mainly by the specimen heating temperature and is little affected by the grade of steel.

Table 2 gives the results of extraction of electrolytic and metallurgical hydrogen at various heating temperatures.

Column 1 gives the amount of electrolytic hydrogen* separated from various steel grades by 1 h of polarization in sulfuric acid, column 2 gives the amount of metallurgical hydrogen extracted from the original specimens while column 3 gives the amount of metallurgical hydrogen separated from specimens in 5 h of heating. At temperatures of 200 and 400°C, a relatively small amount of metallurgical hydrogen is separated during the period of time needed to extract the electrolytic hydrogen. An increase in extraction temperature increases the error associated with the separation of metallurgical hydrogen.

For a comparison of the rates of gas removal from various steels, Fig. 3 gives the dependence of the amount of extracted hydrogen on the duration of its separation in vacuum at 400°C. The duration of extraction of electrolytic hydrogen was approx. the same for all steels whereas the amount of adsorbed gas and the extraction rate depend on the steel grade. Additional tests showed that even when the specimens are saturated with hydrogen (during cathode polarization for 10 h) the duration of extraction is for practical purposes the same as that given in Table 1.

After heating, each specimen was transferred into the induction furnace and melted. During this process the same amount of hydrogen was extracted as during the melting of the initial specimens. Thus, the method of vacuum melting ensures the complete extraction of the electrolytic hydrogen from steel.

*The extraction time in columns 1 and 2 is given in Table 1.

TABLE 2. Amount of Hydrogen (cm³/100 g) Separated under Various Heating Conditions

Temp. °C	Temp. °C			30KhGSA			U8			A12		
	1	2	3	1	2	3	1	2	3	1	2	3
200	0,6-0,7	0,05	0,1	0,4-0,5	0,03	0,07	1,7	0,03	0,08	3,5-3,8	0,08	0,1
400	0,6-0,7	0,11	0,15	0,4-0,5	0,05	0,15	1,7	0,04	0,1	3,5-3,8	0,13	0,25
600	0,6-0,7	0,17	0,40	0,4-0,5	0,11	0,36	1,7	0,1	0,4	3,5-3,8	0,3	0,40

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