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Abstract

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CHEMISTRY

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SOLUBILITY OF HYDROGEN IN LIQUID NIOBIUM

(Presented by Academician B. E. Paton on 19 V 1965)

In terms of the nature of its interaction with hydrogen, niobium belongs to the group of exothermic occluders. The solubility of hydrogen in solid niobium has been studied (¹⁻⁴) at comparatively low temperatures (almost 1000° below the melting point). The results obtained in these works are in poor agreement with one another. The magnitude of the thermal effect of dissolution according to the data of works (¹⁻⁴) ranges from 16,050 to 21,360 cal/mole H₂ (67,100 to 89,200 J/mole H₂).

There are no data in the literature on the solubility of hydrogen in liquid niobium. At the same time such information is extremely necessary for solving the problem of obtaining pure niobium and refractory alloys based on it.

To study the solubility of hydrogen in liquid niobium, the present work used the method of crucibleless melting, in the suspended state, of small metal samples in a stream of a mixture of inert gas with hydrogen (⁵). After equilibrium had been established between hydrogen in the gas phase and in liquid niobium, the sample was dropped into a massive copper mold with a gap in the form of a narrow wedge. This ensured reliable fixation of the hydrogen dissolved in the metal at the experimental temperature. The temperature of the molten niobium sample was measured with a TsEPIR-010 optical pyrometer, with the readings recorded on the tape of a self-recording instrument. Hydrogen in the metal was determined in 3 samples cut from the thinnest part of the wedge-shaped specimen. The hydrogen content in the wedge-shaped specimen was taken as the arithmetic mean of three determinations in the cut-out samples. Hydrogen analysis in niobium was carried out by the vacuum-heating method at 1673° K.

For the investigation, niobium of double electron-beam remelting and the gases argon, helium, and hydrogen, carefully purified of impurities, were used.

The solubility study was carried out at hydrogen partial pressures from 7 to 62 mm Hg in the temperature interval 2873-3093° K.

In view of the fact that under our conditions of melting in the suspended state it was impossible to reproduce the experimental temperature results exactly, all

Fig. 1. Dependence of the solubility of hydrogen in liquid niobium on temperature: S_{50} —solubility at a gas pressure of 50 mm Hg, $\text{cm}^3/100 \text{ g}$

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experiments were carried out in four temperature intervals: I $-2873-2923^\circ \text{ K}$, II $-2931-2965^\circ \text{ K}$, III $-2982-3014^\circ \text{ K}$, IV $-3073-3093^\circ \text{ K}$.

For the mean temperature of each temperature group of experimental points, the exponent of the solubility-isotherm equation, “ n ,” was determined. Despite the rather considerable temperature interval within each group, the exponent proved to be close to the value 0.5 (respectively 0.53; 0.52; 0.51 and 0.47). In temperature group I there were 9 experimental points, in II —7, in III —4, and in IV —8.

The dependence found for the solubility of hydrogen in liquid niobium on temperature is presented in Fig. 1 in the coordinates $\lg S_{50} - 10^4/T^\circ \text{ K}$. All the experimental data obtained were reduced to a hydrogen pressure in the gas phase of 50 mm Hg by means of the isotherm equation:

$$S_{50}/S_i = (50/P_i)^{0.5},$$

The straight line in Fig. 1 is drawn through points representing the mean square solubility of hydrogen in individual experiments of the three temperature groups.

The temperature dependence presented is analytically expressed by the following semiempirical equation:

$$\lg S_{50} = 1620/T + 0.993,$$

where S_{50} is the solubility of hydrogen at a gas pressure of 50 mm Hg, $\text{cm}^3/100 \text{ g}$, and T is the absolute temperature, $^\circ \text{ K}$.

The correlation coefficient of this dependence, determined from all individual experimental points, is 0.74.

The solubility of hydrogen in liquid niobium, as in solid niobium, decreases with increasing temperature. The rate of decrease of solubility in the liquid metal is lower than in the solid. According to our determinations, the heat of solution of one mole of hydrogen in liquid niobium is 14,800 cal (62,000 J).

Fig. 1. Dependence of the solubility of hydrogen in liquid niobium on temperature: S_{50} —solubility at a gas pressure of 50 mm Hg, $\text{cm}^3/100 \text{ g}$

At the melting point, the solubility of hydrogen changes discontinuously. The solubility of hydrogen in liquid niobium is higher than in solid niobium. The magnitude of the jump, determined by extrapolating our data and the data of

works ⁽³⁾ and ⁽⁴⁾ to the melting point of niobium, is equal to 2.67 and 7.71, respectively.

Taking into account that the heat of solution of hydrogen in metals depends only weakly on the state of aggregation and that the solubility jump in metals is usually $\sim 2-3$ (with the exception of aluminum), it may be assumed that the more reliable value of the heat of solution of hydrogen in solid niobium is 16,050 cal/mole H₂ ⁽³⁾.

It is evident from the figure that the second group of points (2931-2965°K) falls outside the general regularity of the behavior of hydrogen solubility with changing temperature. Deviations from the square-root law were not found in these experiments; nevertheless, all seven experimental points formed a compact group below the straight line expressing the general regularity. Similar anomalies are also observed in other systems. In particular, in the liquid iron-hydrogen system in the region of 2300°K, and in the liquid titanium-hydrogen system at temperatures above 2700°K. The liquid metal is, in all probability, responsible for these deviations. Work is currently under way to determine the causes of these anomalies.

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Note: Figure translations are in progress. See original paper for figures.

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