04 OCT. 2016

Magnetic properties of solutions of hydrogen in nickel-iron alloys

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Fiz. Tverd. Tela (Leningrad) 20, 2680-2686 (September 1978)

The behavior of the spontaneous magnetization σ_0 at O^oK and of the parameter of the fcc metal sublattice at 83^oK was investigated for solutions of hydrogen with the atomic ration H/Me \gtrsim 1 based on the Ni-Fe series of fcc alloys. The dependence of the Curie point $T_c = T_c(P_{H_2})$ was determined for the 67.5 at.% Fe Invar alloy in the range $P_{H_2} \le 50$ kbar. Samples of Me-H were prepared by quenching after exposure of an investigated alloy foil to hydrogen at pressures up to 70 kbar.

PACS numbers: 75.50.Bb

An isomorphous $\gamma_1 \rightarrow \gamma_2$ transition takes place in nickel at high hydrogen pressures; here, γ_1 and γ_2 are the interstitial solid solutions with low and high amounts of hydrogen in the fcc crystal lattice of nickel.¹ The line corresponding to this transition in the $T - P_{H_2}$ phase diagram of the Ni-H system terminates at a critical point. Alloying of nickel with iron lowers the critical temperature.^{2,3} When the iron concentration is in excess of 20-40 at.%, the solubility of hydrogen in the $P_{H_2} \leq 50-70$ kbar range is a continuous function of the hydrogen pressure at temperatures above the room value.^{2,4,5}

We studied earlier the influence of the hydrogen pressure up to 20 kbar on the Curie point T_C of nickel,^{6,7} of Ni-Fe alloys containing 5, 10, and 15 at.% Fe (Ref. 2), and of two Invar Ni-Fe alloys with 64 and 67.5 at.% Fe (Ref. 4). We also studied⁵ the influence of dissolved hydrogen on the spontaneous magnetization of Fe_{65} (Ni_{1-X}-Mn_X)₃₅ alloys at the atmospheric pressure.

The present paper reports an investigation of the magnetization of hydrogen solutions in disordered fcc Ni-Fe alloys containing 10, 20, 40, 60, 66.1 and 67.5 at.% Fe. The Ni-Fe-H samples were prepared by quenching to T < 273 K after hydrogen saturation for several hours at T = 523 K and hydrogen pressures up to 70 kbar. In the case of the 67.5 at.% Fe alloy we obtain the dependence of the Curie point on the hydrgeon pressure in the range $P_{H_2} \le 50$ kbar. Moreover, we found the dependence of the parameter of the fcc metal sublattice on the hydrogen content for solutions of hydrogen in alloys with 20 and 67.5 at.% Fe at P = 1 atm and T = 83 K.

1. PREPARATION OF SAMPLES AND EXPERIMENTAL METHOD

The alloys were prepared in an argon-filled induction furnace from nickel of the NO grade and carbonyl iron. The resultant ingots were subjected to homogenizing annealing at T = 1400 % for 10 h, followed by quenching in water. They were then cold-rolled into a foil ~ 0.2 mm thick, annealed (to remove mechanical stresses) at T =1400 % for 15 min, and once again quenched in water. Samples containing 66.1 and 67.5 at.% Fe were prepared from the same foil as in Refs. 4 and 5.

The magnetization was determined for samples of 20-30 mg mass, which consisted of 10×1.5 mm foll strips. The magnetization was determined by the induc-

tion method using ~0.01 sec magnetic field pulses⁸ at temperatures from 80 to 220 K. Measurements at higher temperatures were prevented by the decomposition of the NI-Fe-H samples into a metal and molecular hydrogen, which occurred at a significant rate beginning from ~240 K (Refs. 4 and 5).

The Curie points T_C of the alloy with 67.5 at.% Fe were determined by the differential transformer method from anomalies of the isobars of the temperature dependence of the initial magnetic permeability μ_0 (Ref. 9). A ring with an outer diameter of ~4 mm cut from the foil of the investigated alloy was used as the transformer core. The temperature of the sample was measured with a Chromel-Alumel thermocouple protected from the action of hydrogen. The pressure was determined to within ± 4 kbar and the temperature to within ± 15 %. The corrections to the thermocouple readings due to the action of high pressures were ignored.

Methods for preparing Ni-Fe-H solutions metastable at P = 1 atm by quenching after saturation with hydrogen at high pressures (up to 70 kbar) were described in Ref. 4, together with the methods used in chemical analysis (to within ~3% for $n \ge 0.02$) and x-ray diffraction at atmospheric pressure (use was made of the Co K α radiation).

2. EXPERIMENTAL RESULTS

Dependences of the Volume on the Hydrogen Concentration in Alloys with 20 and 67.5 at.% Fe

Figure 1 shows the results of determination, at atmo-



FIG. 1. Dependences $\Delta V_0 = V(n) - V(0)$ obtained at T = 83°K for the alloys with different iron concentrations (at%Fe): 1)20; 2) 67.5.

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0038-5654/78/09 1548-05 \$02.40



FIG. 2. Dependences of the spontaneous magnetization σ_0 at 0°K and atmospheric pressure on the hydrogen content in the Ni-Fe alloys with different iron concentrations (at,%Fe): 1) 10; 2) 20; 3) 40; 4) 60; 5) 66.1; 6) 67.5. The chain line gives the calculated dependence for Ni.

spheric pressure and T = 83 K, of the dependence ΔV_0 = V(n) - V(0), where V(n) and V(0) are the volumes of the unit cells of the NI-Fe alloys with the hydrogen concentration n and without hydrogen, respectively. The chain line represents the dependence $\Delta V_0(n)$ at T = 293 %, satisfied well by a number of Me-H solutions with the fcc sublattice of palladium (transition metal) and by its alloys with Ir, Au, Ag, Pt, Cu, as well as by Ni-Cu alloys.¹⁰ We can see that the dependence $\Delta V_0(n)$ of the NI-Fe alloy system deviates considerably from that given by the chain line but its nature is the same as for the other alloys mentioned above: in the range n < 0.8 the slope $\beta =$ $(\partial/\partial n) \Delta V_0(n)$ is almost constant (and it amounts to $\beta \approx$ 9.5 $Å^3$), whereas in the range n > 0.8 it decreases strongly (to $\beta \sim 2.5 \text{ Å}^3$). It should be noted that the deviation of the dependence $\Delta V_0(n)$ obtained in the present study from that given in Ref. 10 is not due to the difference between the temperatures (83 and 293°K) at which the measurements were carried out: the values of ΔV_0 for the alloy with 20 at.% Fe and n = 0.89, measured at 83 and 220 %, agree within the experimental error of $\delta(\Delta V_0) \sim 0.5 \text{ Å}^3$, which is mainly due to the width of the diffraction lines of the Ni-Fe alloys and solutions of hydrogen in these alloys.

Dependence $\sigma_0(n)$

The temperature dependence of the magnetization of nickel includes the spin-wave and Stoner terms.¹¹ We may assume that this is also true of our NI-Fe-H samples. The spontaneous magnetizations σ_0 at T = 0% found by extrapolation of the experimental values in accordance with the laws T² and T^{3/2} agree within the limits of the experimental error, which is 5%.



FIG. 3. Dependences of the Curle points of the alloy with 67.5 at % Fe on the pressure in a hydrogen atmosphere (the points denoted by 1 are our results and those denoted by 2 are taken from Ref. 4) and in an inert medium (the points denoted by 3 are taken from Ref. 4).

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It is clear from Fig. 2 that in the case of the alloys with 10, 20, and 40 at.% Fe the zero-point magnetization σ_0 decreases on increase of n and in the range n \notin 0.8 the dependence $\sigma_0(n)$ is nearly linear, whereas for n > 0.8 there is a small but systematic deviation of σ_0 in the direction of higher values. The dependence $\sigma_0(n)$ for the alloy with 60 at.% Fe is nearly linear up to n = 1. The behavior of σ_0 in the case of the alloys with 66.1 and 67.5 at.% Fe is more complex. The magnetization σ_0 of the alloy with 66.1 at.% Fe depends weakly on the hydrogen concentration up to n ~ 0.5 and then begins to fall. The value of σ_0 of the alloy with 67.5 at.% Fe rises with the hydrogen concentration in the range of small values of n, reaches a maximum of ~ 1.85 $\mu_{\rm B}$ /atom at n ~ 0.3, and then decreases monotonically.

Dependence $T_{C}(P_{H_2})$ for the Alloy with 67.5 at.%

Figure 3 gives the results of the determination of the Curle point of this alloy as a function of pressure in an inert medium and in hydrogen. We can see that in an inert medium the Curle point T_C of the alloy at pressures up to 20 kbar decreases linearly at a rate $(dT_C/dP)_{in} = -5.05 \pm 0.1^{\circ}$ K·kbar (Ref. 4). It is reported in several papers (see, for example, Ref. 12) that in the case of the Ni-Fe Invar alloys the dependence $T_C(P)$ in an inert medium is nearly linear (dashed line in Fig. 3) even at higher pressures.

The increase in the hydrogen concentration in the alloy on increase of the hydrogen pressure causes the dependence $T_{C}(P_{H_2})$ to deviate from that observed for an inert medium in the direction of higher values of TC. In the range PH, > 15 kbar the Curie points of the Ni_{0.325}Fe_{0.675}-H solution begin to rise, reaching ~700°K at P_{H_2} = 50 kbar. The second column of Table I gives the values of the hydrogen concentration n on the line representing the Curle points of the alloy with 67.5 at.% Fe at pressures $P_{H_2} = 20$, 35, and 51 kbar. The hydrogen content was determined for samples prepared by quenching after heating at $T = T_C(P_{H_2})$ for 2 h, which was much longer than the time needed for the saturation of the alloy with hydrogen to the equilibrium concentration estimated from the cessation of the time drift of the Curie point of the alloy after establishment of the required pressure.⁴ In the range of pressures where the solubility of hydrogen in the alloy rose most rapidly (~25-35 kbar), the dependence $T_{C}(P_{H_2})$ of the Ni_{0.325}Fe_{0.675}-H solution was also steepest.



FIG. 4. 1) Slopes d_{σ_0}/dn in the range n < 0.8 obtained for alloys with ≤ 60 at % Fe (compare with Fig. 2); 2) dependence of σ_0 of the Ni-Fe alloys on the iron concentration. The values of $\partial \sigma_0/\partial n$ and σ_0 are in units of $\mu_B/atom$ of the Ni-Fe alloy; n is dimensionless.

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3. DISCUSSION

Spontaneous Magnetization $\sigma_0(n)$

It is clear from Fig. 2 that the zero-point magnetization σ_0 of the alloys with ≤ 60 at.% Fe decreases approximately linearly with n in the range n < 0.8. We recall that a composition n ~ 0.8 is distinguished by the fact that, for all the interstitial hydrogen solutions based on the fcc sublattice of the transition metal, there is a change in the dependence $\Delta V_0(n)$ in the range n > 0.8. The reason for this change is not yet clear. It has been suggested that in the solutions with n > 0.8 the hydrogen begins to occupy not only the octahedral but also the tetrahedral interstices in the investigated metals.⁷

Figure 4 gives the slopes $\partial \sigma_0 / \partial n$ found by the leastsquares method in a linear approximation of the experimental dependences $\sigma_0(n)$ in the range n < 0.8 for the alloys containing ≤ 60 at.% Fe. It is clear from this figure that the values of $|\partial \sigma_0 / \partial n|$ increase monotonically and approximately linearly on reduction of the iron content of the alloys. Extrapolation to nickel gives $\partial \sigma_0 / \partial n = -0.63 \ \mu_B$. atom⁻¹. The dependence $\sigma_0(n)$ with this slope for nickel is represented by the chain line in Fig. 2. According to the neutron-diffraction data, 13 the composition of nickel hydride γ_2 coexisting with the phase γ_1 at atmospheric pressure and room temperature is given by $n_{V_n}^{\min} = 0.6 \pm 0.1$. It is clear from Fig. 2 that this composition is close to the limit at which the ferromagnetic order can still exist. Our γ_2 samples of Ni-H with $n \ge 0.8$ are paramagnets at $T \ge$ 83°K. Thus, measurements at lower temperatures are needed before we can say whether the nickel hydride in question is ferromagnetic.

Nickel is described satisfactorlly by the band ferromagnetism model.¹⁴ Near T = 0% it can be regarded as a strong band ferromagnet. The concentration dependence of σ_0 of the NI-Fe alloys containing up to 60 at.% Fe is nearly linear, agrees with the Slater-Pauling curve, and has the slope $\vartheta \sigma_0 / \vartheta x_{Fe} \approx 2.1 \ \mu_{B}/atom = 1.05 \ \mu_{B}/electron$ (see Ref. 15 and also our data in Fig. 4; here, x_{Fe} is the atomic fraction of iron in the Ni-Fe alloy), i.e., it is described satisfactorily by the rigid band model of strong ferromagnets.

The hydrogen occupying interstices in the metals at the ends of the d periods of the Mendeleev table, particularly in the Ni-Fe alloys, should not form bound states below the Fermi level and should give up its s electron to the conduction band of the metal.¹⁶ Since, as pointed out above, the Ni-Fe alloys containing ≤ 60 at.% Fe can be regarded as strong band ferromagnets, the introduction of hydrogen considered in the rigid band approximation gives $\partial \sigma_{1}^{-}/\partial n = -1 \mu_{\rm B}/$ atom. It is clear from Fig. 4 that the experimental values of $\partial \sigma_0 / \partial n$ range from -0.6 for the alloy with 10 at.% Fe to $-0.4 \mu_B/atom$ for the alloy with 60 at.% Fe.

Consequently, the hydrogen deforms strongly the energy band structure of the Ni-Fe alloys, which may be expected on the basis of the calculations of the energy band structures of hydrides of some transition metals, particularly of nickel and its closest analog, which is palladium.17,18 According to these calculations, an increase in the number of protons at interstices in these metals deforms the energy spectrum in such a way that the number of states below the Fermi level increases and, consequently, only some of the electrons [~0.4-0.1 electrons per proton for Pd and NI (Ref. 17)] are supplied by the hydrogen atoms to the conduction band filling the states above the Fermi level and the rest fill additional levels below the Fermi energy. We can see that the sign and order of magnitude of the slopes $\partial \sigma_0 / \partial n$ obtained for our alloys are in agreement with these calculations.

The hydrogen dissolved in metals expands considerably the crystal lattice. Since σ_0 of the Ni-Fe alloys, particularly those in the Invar range of concentrations, is strongly volume-dependent, 19 it would be interesting to estimate the role of the volume effect in the change of σ_0 resulting from the introduction of hydrogen into these alloys. We recall that at $P \leq 20$ kbar the deviation of the pressure dependence of the Curie points of the Ni-Fe Invars with 64 and 67.5 at.% Fe in the hydrogen atmosphere from the corresponding pressure dependence in an inert atmosphere is mainly due to the volume effects.⁴ Therefore, we shall assume that at least for n < 1 we can separate the volume effect from the total change in σ_0 on increase of the hydrogen concentration. Then, the contribution of the volume changes to the initial slope of the dependences $\sigma_0(n)$ is

$$\frac{\partial z_{0}^{V}}{\partial n}\Big|_{n=0} \approx \left(\frac{\partial z_{0}}{\partial V}\right)_{10} 3 \approx -\left(\frac{\partial z_{0}}{\partial P}\right)_{10} \frac{3}{kV},$$

where k is the compressibility; V is the volume of a unit. cell of the Ni-Fe alloy at T = 0 %; the subscript "in" denotes an inert medium.

Using the values of σ_0 and β obtained in the present study and then taking $(1/\sigma_0)(\partial\sigma_0/\partial P)_{in}$ from Refs. 20 and and 21, the compressibility k at T = 288 % from Ref. 22, and the unit-cell volume V at 288 °K from Ref. 23 (reduced to T = 0 % using the thermal expansion coefficients from Ref. 24), we find that in the case of the samples containing 0, 40, and 60 at.% Fe the values are $\partial\sigma_0^V/\partial n|_{n=0} \approx 0.1$, 0.3, and 2 $\mu_B/atom$, respectively. We can see that the contribution of the volume changes to $\partial\sigma_0/\partial n_{n=0}$ is considerable even for nickel and for the alloy with 40 at.% Fe

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TABLE I.	Properties of	Investigated	Samples
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*Fo	n	P, kbar	—{n	т <mark>п</mark> , °К	T [₽] _C , °K	TP + TC K	TC, K	Т <mark>С,</mark> "К
0 675 0.675 0.675 0.1	0.041 0.735 0.99 0.395	20 35 51 21.5	0.018 0.136 0.183 0.1	58 285 365 115	2 90 160 _5	60 375 525 	71 365 520 145	55

Note: The second column gives the values of n at $P_{H_2} = P$ from the third column. The temperature is $T = T_C(P_{H_2})$.

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It is of the same order of magnitude as the deviation of the observed value of $\partial \sigma_0 / \partial n$ from $\partial \sigma_0^T / \partial n = -1 \mu_B / a tom$. However, already for the alloy with 60 at.% Fe the value of $\partial \sigma V / \partial n$ is so large that the difference between it and the experimental value $\partial \sigma_0 / \partial n$ cannot, in principle, be compensated even by assuming that the increase in the electron density alters the magnetization, as in the rigid band model:

$$\Delta \frac{\partial \sigma_0}{\partial n} \Big|_{n=0} = \frac{\partial s_v^r}{\partial n} + \frac{\partial s_v^n}{\partial n} \Big|_{n=0} - \frac{\partial s_0}{\partial n} \approx -1 + 2 + 0.4 = 1.4 \mu_{\rm B}/{\rm atom},$$

Moreover, there are no grounds for postulating that the value of $\partial \sigma_0^{V/} \partial n$ remains constant as the hydrogen concentration is increased, especially as it is known that the dependence $\sigma_0(n)$ for the alloy with 60 at.% Fe is nearly linear (Fig. 2).

It follows that even in the $n \ll 1$ case the influence of hydrogen on σ_0 of the nickel-iron alloys cannot be described by a sum of two terms associated with changes in the electron density and in the volume.

Nevertheless, it is possible to introduce some elements of order and even a semiquantitative description into the chaos of the experimental data resulting from the above conclusion.

The concentration dependences of the spontaneous magnetization of the alloys with 66.1 and 67.5 at.% Fe are basically similar when iron is replaced with nickel (Fig. 4) and when hydrogen is introduced (Fig. 2): at some concentration the value of σ_0 reaches its maximum value $\sigma_0^{max} \approx 1.86-1.9 \ \mu_B/atom$ and then begins to fall. In the case of the alloys with $x_{Fe} \leq 0.6$ the magnetization decreases linearly on replacement of Fe with Ni on increase of n. We shall introduce a conversion factor ξ relating, for each specific Ni-Fe alloy with $x_{Fe} \leq 0.6$, the values of n and Δx_{Fe} :

$$\Delta x_{\rm Fe} = \frac{\left(\frac{\partial \sigma_0}{\partial n}\right)}{\left(\frac{\partial \sigma_0}{\partial x_{\rm Fe}}\right)} n = -\xi n. \tag{1}$$

The expression (1) describes simply the experimental observation that the dependence $\sigma_0(n)$ of such alloys can be obtained approximately from $\sigma_0(x_{Fe})$ by a suitable change of the concentration scale. Extrapolation of the dependence (1) to the alloys with $x_{Fe} = 0.661$ and 0.675 gives $\xi \approx 0.185$ (Fig. 4). If we use this value of ξ and assume that the curve $\sigma_0(x_{Fe})$ reaches its maximum at $x_{Fe}^{max} = 0.62$ (Fig. 4), we find that the positions of the maxima of the dependences $\sigma_0(n)$ for the alloys with $x_{Fe} = 0.661$ and 0.675 are, respectively, $n_{0.661}^{max} \sim 0.1$ and $n_{0.675}^{max} \sim 0.3$, in satisfactory agreement with the experimental results (Fig. 2).

Dependence TC(PH2)

If we assume that the relationship (1) between σ_0 of the alloys with and without hydrogen applies also to T_C and $(dT_C/dP)_{in}$, we can describe the behavior of ΔT_C^{exp} , which is the difference between the values of T_C in hydrogen and in an inert medium at the same pressure (Fig. 3). If this approach is adopted, we find that $\Delta T_C(P)$ consists of two terms, one of which is

$$\Delta T_{C}^{n} = T_{C} \left(x_{Fe} - \xi n \right) - T_{C} \left(x_{Fe} \right)$$

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associated with the change in T_C due to introduction of hydrogen without pressure, and the other is

$$\Delta T_{\rm C}^{P} = \left\{ \left(\frac{dT_{\rm C}(x_{\rm Fe} - \xi n)}{dP} \right)_{\rm in} - \left(\frac{dT_{\rm C}(x_{\rm Fe})}{dP} \right)_{\rm in} \right\} P,$$

due to the different pressure dependences of the Curie points of the alloys with and without hydrogen. The results of the calculations are given in Table I. The values of T_C of the Ni-Fe alloys are taken from Ref. 25 and the values of $(dT_C/dP)_{in}$ are obtained from Ref. 26.

It is clear from Table I that the calculated values of $\Delta T_C^R + \Delta T_C^R$ are in satisfactory agreement with ΔT_C^{exp} . It should be noted that at high pressures the contribution ΔT_C^R to $\Delta T_C^n + \Delta T_C^p$ is not small and, therefore, the agreement of the latter quantity with ΔT_C^{exp} confirms the applicability of Eq. (1) to the dependences of T_C and of $(dT_C/dP)_{in}$ of the Ni-Fe invars on the hydrogen content. Consequently, the existence of the relationship (1) allows us to determine the tendency of change represented by $[dT_C(n)/dP]_{in}$, which would be very difficult to find experimentally.

The last column of Table I gives the results of a similar calculation for a non-Invar alloy with 10 at.% Fe, for which the dependence $T_C(P_{H_2})$ is given in Ref. 2. The values of ΔT_C^{exp} and ΔT_C^{exp} were calculated using $(dT_C/dP)_{1n}$ taken from Ref. 12. The results (Table I) indicated that Eq. (1) described satisfactorily the behavior of $T_C(P_{H_2})$ of this alloy as well.

We shall conclude by noting once again the interesting fact that in the $n \ll 1$ range the behavior of the dependence $T_C(n)$ of the Invars can be described on the assumption that the main effect of the introduction of hydrogen is the expansion of the crystal lattice.⁴ In this case the contribution of the volume changes to ΔT_C is⁴

$$\Delta T_C^{\gamma} \approx -\frac{\left(\frac{dT}{dP}\right)_{in} \frac{3n}{kV}}{1 + \left(\frac{dT}{c}/dP\right)_{in} \frac{3a}{k}},\tag{2}$$

where α is the linear expansion coefficient of the alloy at $T \sim T_C$. It is clear from Table I that the value of ΔT_C^V calculated using the above expression is in good agreement with ΔT_C^n for the alloy with 67.5 at.% Fe. The sign and order of magnitude of ΔT_C^V agrees with ΔT_C^n also of the alloy with 10 at.% Fe [in this case n is not small, but, because the quantities occurring on the right-hand side of Eq. (2) vary slowly in the range $\xi_n \sim 0.1$ for this alloy, the above estimate can be obtained using the values of $(dT_C/dP)_{in}$ and k for the alloy with 5 at.% Fe].

It follows that in the range of small values of n the behavior of the Curie points of the γ solutions of the Ni-Fe-H type containing > 10 at.% Fe is dominated by the volume effects. The behavior of the Curie points of these alloys at high hydrogen concentrations is, like the behavior of σ_0 , more complex. However, the dependences $\sigma_0(n)$, T_C(n), and dT_C(n)/dP can be described semiquantitatively using the relationship (1) and the corresponding dependences of these quantities on the composition of the Ni-Fe alloys. It is not yet clear why this relationship applies. Further information on this subject can be obtained by investigating the magnetic properties of hydro-

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gen solutions in other nickel-base alloys.

The authors are grateful to I. A. Potapov and A. I. Amelin for their help in the preparation of the experiments.

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Translated by A. Tybulewicz

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