PHYSICAL REVIEW B VOLUME 39, NUMBER 2 15 JANUARY 1989-I

Diffusivity and solubility of Ni (^{63}Ni) in monocrystalline Si

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(Received 29 August 1988)

Diffusion of Ni atoms in P-doped Czochralski-grown (Cz) monocrystalline Si has been studied using the ^{63}Ni radiotracer and autoradiographic and sectioning techniques. The diffusivity of Ni in P-doped Cz Si is described by $D=6\times10^{-4}\exp[(-0.76\text{ eV})/kT]$ cm^2/sec over the temperature range of 220–540 °C. The solubility of Ni in Si at 800 °C is about 10^{16} atoms/cm^3. At low temperatures, the solubility is uncertain due to the limit of tracer detection. A critical examination has been made on the published values of Ni diffusion in Si which show a scatter of 10 orders of magnitude among the various investigations. The investigations which report low-diffusion coefficients and large activation energies are suspect due to the problems encountered with the surface conditions and limitations imposed on Ni detection by low solubility and fast diffusion. Our data on Ni diffusion in Si substantiate an interstitial mechanism without intervention of the native defects in Si.

I. INTRODUCTION

The impetus of the extensive research on transition-metal silicides in the last decade came from their applications in very-large-scale–integration (VLSI) technology as low-resistivity gates, interconnections, and contacts. The criteria for selecting a silicide depend not only on its physical properties such as conductivity and Schottky barrier to Si but also on the behavior of the metal atoms as impurities in Si. Many transition metals probably have deep-lying acceptor levels in Si (Refs. 1 and 2) and show high diffusivities in Si (Ref. 3). Their application in Si devices may therefore cause problems of current leakage and changes in other electrical characteristics such as threshold capacitance. A general solution to the problem has been the use of diffusion gettering to remove the unwanted metal impurities by techniques such as oxidation or heavy doping on the backside of the Si substrate which introduces Si interstitials and results in "kick-out" of the substitutional metal impurities. If the transition-metal impurity is already an interstitial species no such treatment is required as it can be easily gettered by a sink. The gettering of Cu or Au through a silicon wafer by employing an Al sink on the backside was shown in a Rutherford backscattering (RBS) study by Thompson *et al.*[4] Since the Cu or Au atoms could reach the Al sink by diffusing across the entire thickness of the Si wafer, a distance of the order of ∼200 μm, at relatively low temperatures (∼500 °C) in a few hours, the diffusion has to be extremely fast and in all probability the atoms move as interstitials.

Thompson *et al.*[5] have extended the gettering experiments with Ni atoms diffusing in Czochralski (Cz) -grown Si monocrystalline wafers using the same backside Al-sink technique. In that study the effects of several variables such as temperature, the Si substrate thickness and annealing times were investigated and the product of the diffusion coefficient D and the concentration C for the Ni atoms being transported in the Si lattice was determined. It was described by $DC=8\times10^{22}\exp[(-2.16\text{ eV})/kT]$ atoms/cm sec. Although, the individual values of D and C could not be measured, an estimate of the former could be made indirectly by making certain assumptions on the Ni solubility and allowance for the Ni trapping by the P-dopant atoms. Despite accounting for the Ni-P trapping, which is supposed to slow down the Ni interstitial diffusion, the diffusion coefficient of Ni could be expressed by $D=6.3\times10^{-4}\exp[(-0.76\text{ eV})/kT]$ cm^2/sec. The diffusivity is on the high side when it is compared to the recent compilation[6] of data on diffusion coefficients of Ni in Si which shows an unusual scatter of 10 orders of magnitude among various investigators. Consequently, no diffusion mechanism can be assigned unequivocally to diffusion of Ni atoms in the Si lattice nor can these data be used for the VLSI applications alluded to earlier.

In view of the preceding situation, we have performed direct measurements of the diffusion coefficient of Ni in Si at low temperatures employing the ^{63}Ni radioisotope. We have used the serial-section technique to profile the penetration of ^{63}Ni into P-doped, monocrystalline, Czochralski-grown (Cz) Si wafers in the 300–800 °C range. We have also used the ^{63}Ni radiotracer in the gettering or percolation mode without the Al sink on the backside of the wafer. We found that the (unpolished) backside surface itself was an adequate sink for the arriving ^{63}Ni isotope, due to the high sensitivity of the autoradiographic technique. With the serial-sectioning technique we determined absolute concentration profiles of ^{63}Ni atoms in the Si sample. It was not possible to extract diffusion coefficients from these profiles due to the complexity of a very fast Ni diffusion in Si and a vanishingly small Ni solubility at the low temperatures investigated. However, our ^{63}Ni percolation experiments turned out to be capable of providing meaningful diffusion coefficients. Consequently we can describe the diffusion of Ni in Si with a significantly better precision in the 220–540 °C temperature range. In light of the new results, we have reexamined the various diffusion investigations reported in the literature[6] in terms of reliability and some of the unsuspected experimental difficulties which might have been encountered.

II. EXPERIMENTAL PROCEDURES AND RESULTS

The Si monocrystalline specimens used in this study were Cz-grown, ⟨111⟩ oriented, *n*-type (P-doped; 10 Ω cm) wafers of 1 in. diameter and 280 μm thickness. The radiotracer used was ^{63}Ni isotope which was received as NiCl in 0.5*M* HCl solution from New England Nuclear DuPont Biotechnology Division. The isotope is a β-emitter of 67-keV energy with a half-life of 92 y. In order to measure diffusion of ^{63}Ni in Si we used two different techniques which are described separately below.

A. Indepth profiling of ^{63}Ni tracer in P-doped Cz Si

The first technique employed was the well-known serial sectioning method in which the penetration of the radioactive tracer is profiled. The details of this technique have been described elsewhere.[7,8] Initially the ^{63}Ni isotope was flash evaporated from a Ta boat in a vacuum of 5×10^{-5} Torr. Later, it was electroplated from a dimethyl sulfur oxide (DMSO) solution, which resulted in an adherent deposit. Following annealing of the samples in evacuated quartz ampules, we used a dc Ar-beam sputtering system for serial sectioning. Our apparatus is similar in design to that of Mehrer and Meier,[9] with the same modifications made by Mundy and Rothman.[10] Like them we also used a beam voltage of 500 V and rotated the sample during sputtering at 15 rpm to ensure homogeneous material removal. A new feature of our apparatus is the watercooling of the rotating sample holder which is performed with the help of a rotating hollow shaft feedthrough (Ferrofluidics Corp., Nashua, New Hampshire) and a rotating union (Deublin Corp., Northbrook, Illinois). The samples were attached to the sampleholder with Wakefield Thermal Compound, without any mask. The sputtered-off material was collected consecutively onto a DuPont Kapton film roll. A shutter was used to interrupt sputtering of the sample rather than turning off the ion beam. Our collection efficiency was about 70%. Ordinarily, counting of low-energy β radiation from the ^{63}Ni isotope requires the liquid-scintillation technique. Fortunately, we were able to count the β activity from ^{63}Ni on the Kapton films directly with a CaF_2-NaI(Tl) solid-state detector supplied by the Bicron Corp., Newbury, Ohio. Although the counting efficiency was only 3%, it was very convenient. The exposed frames upon which the sputtered-off material was deposited were cut from the Kapton film roll. The individual frames of the Kapton film were then mounted on carrier plates which were automatically transported under the CaF_2-NaI(Tl) probe mentioned above and counted in the flat-on geometry. Counting rates were corrected for background.

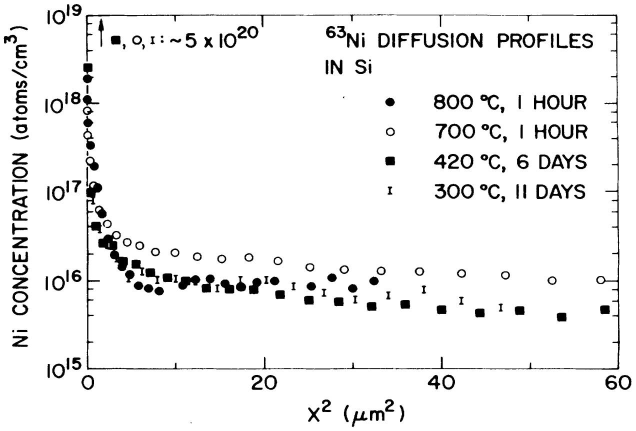

FIG. 1. ^{63}Ni radioactive tracer depth profiles in P-doped Cz Si samples.

In Fig. 1 several ^{63}Ni concentration depth profiles into Si samples are shown. The absolute ^{63}Ni concentrations in these profiles were computed from the specific counting rates (counts/g) in the individual section and the value of the specific activity of the isotope (Ci/g) furnished by the manufacturer. A solubility of $\sim10^{16}$ atoms/cm^3 for Ni occurs at 800 °C which is similar to those reported in the literature.[11,12] Lack of any significant slope in the profiles implies fast diffusion and homogenization of Ni in the Si samples. At temperatures lower than 800 °C a drop in the Ni solubility may be expected as reported by Wiehl *et al.*[12] The reasons why it is not observed in Fig. 1 could be several. Firstly, the material used in this investigation is P-doped Cz Si which can result in substantial trapping of Ni and saturation at 10^{16} Ni atoms/cm^3 as discussed earlier.[4] Secondly, 10^{16} Ni atoms/cm^3 appears to be the limit of detectability of the tracer technique as may be seen in other investigations as well.[11,13] Experiments using neutron-activation analyses of Ni in P-doped Cz Si are in progress to ascertain this. In any event, it is not possible to compute diffusion coefficients for the Ni tracer from the profiles in Fig. 1 due to insignificant slopes.

B. Percolation studies of ^{63}Ni in P-doped Cz Si

Percolation of the tracer is an alternative technique for measuring fast diffusion through thin films and foils.[14] It can be conveniently used for the low-energy β radiation from the ^{63}Ni radioisotope in conjunction with autoradiography. The ^{63}Ni radiotracer was electroplated on the Si samples which were annealed in evacuated quartz ampules as described above. Kodak DR5 x-ray films were used to detect the arrival of the ^{63}Ni tracer on the backside of the Si wafer by diffusion through the entire wafer thickness. As a check, no radioactivity was detected at the back of the specimens incorporated with the ^{63}Ni tracer prior to diffusion. At any temperature, progressively longer annealing times were used to estimate the time at which the tracer could be first detected in the autoradiograms of the back of the samples.

In Fig. 2 the results of the autoradiograms are shown in a plot of $\ln(1/t)$ versus $(1/T)$. The "Δ" symbols mean no detection and thus insufficient tracer penetration through the Si specimen. The "+" symbols mean yes and imply that ^{63}Ni tracer has penetrated through the entire wafer thickness. We have also plotted in Fig. 2 re-

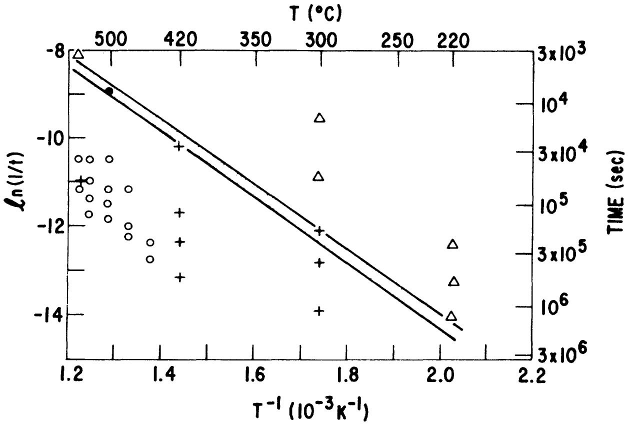

FIG. 2. Percolation of ^{63}Ni radioisotope through 280-μm-thick P-doped Cz Si wafer as detected by autoradiography. t is time in seconds. + means ^{63}Ni is detected and △ means none detected. ○ means Ni detected by RBS in the Al sink in Ref. 5. ● represents the point inferred from the RBS study of Thompson (Ref. 15) (see text). The first detection of ^{63}Ni by autoradiography or Ni by RBS is indicated by the band drawn in the figure.

sults of the RBS study of Thompson *et al.*[5] of backside Ni accumulation in the Al sink, denoted by "○" symbols. All ○ symbols lie substantially below the "yes" measurements for the autoradiograms, implying that the detectability of ^{63}Ni atoms in the autoradiograms is much better. Thompson[15] used the following approach to circumvent the difficulty of the lower sensitivity of RBS measurements. A series of successively thicker Si wafers were used. Ni diffusion after isochronal annealing (20 h, 500 °C) was measured and the amount of Ni collected in the Al sink per unit area was plotted as a function of the wafer thickness. The wafer thickness at which just no Ni would be collected was calculated by extrapolation. A value of 780 μm was found. By use of the expression $x^2=2Dt$, where x is the Si wafer thickness, D the diffusion coefficient, and t the annealing time, we find that our wafer thickness of 280 μm corresponds with an annealing time of 2.58 h at 500 °C within which time no Ni accumulation in the Al sink should occur. This data point (2.58 h, 500 °C) is represented by "●" in Fig. 2. It falls within the band drawn in this figure. This band indicates the time needed for the penetration of a sufficient amount of ^{63}Ni to be detectable by autoradiography as a function of the annealing temperature. Because the wafer thickness is important we assume that the amount of ^{63}Ni penetrated through the entire wafer is only a function of the product Dt. In other words D is proportional to $1/t$ for each temperature T. From Fig. 2 and the Arrhenius equation

$$D=D_0\exp(-E_a/kT)\ , \tag{1}$$

where D_0 is the frequency factor and E_a the activation energy, we can obtain an activation energy of 0.76 eV. To estimate the absolute values of D from the least arrival times of the diffusant at the back of the Si wafers we assumed a Gaussian distribution of the ^{63}Ni. This assumption is justified as follows. The sensitivity of the autoradiograms for low-energy β emissions using Kodak DR5 films is very high: typically, an amount of ^{63}Ni having only a few disintegrations per second per cm^2 can be detected by autoradiography in less than a day in exposure time. So for diffusion times and temperatures represented by the band drawn in Fig. 2 the concentrations of Ni at the backsurface of the wafers was so small that the sample still can be considered to be semi-infinite. The Gaussian distribution using the instant source conditions is given by

$$C(x,t)=\frac{M}{2\sqrt{\pi Dt}}\exp\left[\frac{-x^2}{4Dt}\right]\ , \tag{2}$$

in which C is tracer concentration at time t and depth x into the sample and M is the initial strength of the radiotracer source in atoms per unit area. For the ratio between the concentration 5 μm below the front surface and 5 μm above the back surface we assumed a value between 10^{-2} and 10^{-5}. It is easily seen from Eq. (2) that

$$\frac{d^2}{46t}<D<\frac{d^2}{19t}\ , \tag{3}$$

in which d is the thickness of the wafers and t the penetration time as found from Fig. 2. The result of these calculations is given in Fig. 3, in which the literature data[6] are also given. Our autoradiography results agree remarkably well with the earlier RBS measurements of Thompson *et al.*[5] Furthermore, we extended the temperature range downward significantly. Using Eq. (1) the values for D_0 and E_a for the diffusion of ^{63}Ni in P-doped (10 Ω cm) Cz Si samples are 6×10^{-4} cm^2/s and 0.76 eV, respectively.

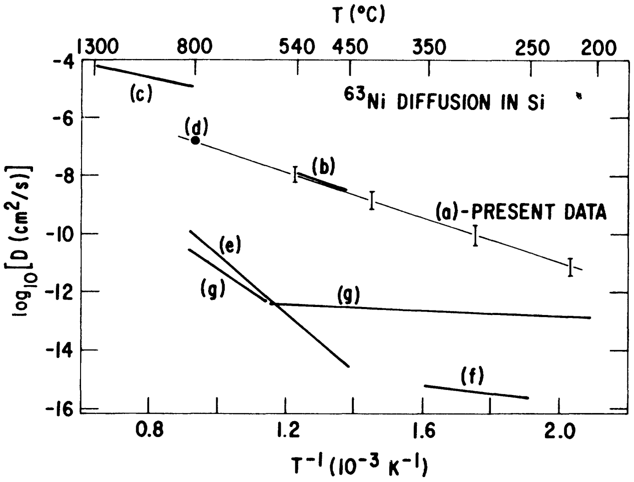

FIG. 3. Arrhenius plots of diffusion coefficients of Ni obtained in this investigation and those reported in the literature. See Table I for the values of the diffusion parameters D_0 and E_a and the references.

III. DISCUSSION

Some general observations can be made on the earlier investigations listed in Table I and shown in Fig. 3. We believe that surface conditions in many of these investigations were not duty controlled or taken into account and may have resulted in unreliable diffusion coefficients. Diffusion measurements of foreign metallic species in Si are difficult and analysis of the data complex as compared to metals, alloys, and even oxides. Particularly, the Ni diffusion in Si is complicated by the vanishingly small solubilities and rather large diffusion coefficients even at very low temperatures. These two factors make the conventional diffusion measurements employing serial-sectioning technique or nuclear absorption very difficult and perhaps impossible. Even at very low temperatures, a large amount of material has to be removed in the sectioning technique, yet there is insufficient diffusant present in the section for any reasonable detection. This situation is reflected in our penetration profiles shown in Fig. 1. A similar quality of ^{56}Co penetration profile may be seen in Ref. 16. It appears that acceptable profiles from which diffusion coefficients can be obtained only at temperatures higher than about 800 °C (where the Ni solubility is high enough) and employing bulk specimens and the grinding technique similar to the investigation of Bakhadyrkhanov *et al.*[17]

In the following we shall discuss the investigations represented in Fig. 3 and investigation (a) of Table I. We divide them into investigations (a)–(d) yielding high diffusion coefficients and investigations (e)–(g) yielding low diffusion coefficients. We shall discuss these two groups separately for their physical significance and reliability.

A. Investigations yielding high diffusivities of Ni in Si

The data in the first group, investigation (a) employing the percolation technique, (b) those of Thompson *et al.*[5], (c) Bakhadyrkhanov,[17] and (d) by Ridgway *et al.*,[18] are characterized by a small preexponential factor $D_0 \leq 2\times10^{-3}$ cm^2/sec accompanied by a small activation energy $Q \leq 0.76$ eV. The discrepancy between our results and those of Bakhadyrkhanov *et al.* might be attributed principally to the differences in the base material. We used P-doped (10 Ω cm) Cz Si, while high-purity Si was used in the former investigation. Some mismatch is seen between the two Arrhenius plots of diffusion coefficients, which is not uncommon between investigations covering different temperature ranges. The mismatch aside, the magnitude of the Arrhenius parameters suggests an interstitial character of Ni in the Si lattice in both pure Si as well as P-doped Si.[19] The diffusion of Ni atoms thus does not require any intervention of native defects in Si such as vacancies or self-interstitials. The possibility of trapping of the interstitial Ni with the P dopant has only a small effect in slowing diffusion as was observed earlier.[5] It is to be noted that despite slowing down, the diffusion of the Ni atoms continues to display an interstitial character as is indicated by the values of the Arrhenius parameters shown in Table I. We mention in passing that we examined our profiles for a "kick-out" mechanism[20] by plotting log (specific activity) against log (depth). A "kick-out" profile is supposed to show a linear part with a slope close to and lower than 1. Since our profiles showed a four times larger slope in the above plot, the possibility of a "kick-out" mechanism is ruled out.

The interstitial nature of Ni in the Si lattice is in fact commensurate with its electrical properties and the growth of Ni silicides. The existence of interstitial Ni in a positive charge state was first reported by Ludwig and Woodbury[21] in an electron paramagnetic resonance (EPR) study. Graff and Pieper[22] subsequently reported a level at $E_v + 0.15$ eV by deep level transient spectroscopy, where E_v is the valence band edge. The Ni level is much lower than other transition metal impurities such as Ti,

TABLE I. Ni diffusion in Si.

Case	Technique	Temp. range (°C)	D or D_0 (cm^2/sec)	E_a (eV)	Ref.
(a)	^{63}Ni: Percolation by autoradiogram in P-doped Cz Si	220–540	6×10^{-4}	0.76	Present data
(b)	Ni: RBS with Al sink in P-doped Cz Si	450–540	6.3×10^{-4}	0.76	5
(c)	^{63}Ni: radioactive analysis in Si	800–1300	2×10^{-3}	0.47	17
(d)	Ni on Si AES[a]	800	1.57×10^{-7}[b]		18
(e)	^{63}Ni: nuclear absorption in *p*-type Si	450–800	10^{3}	4.24	30
(f)	Ni AES in P-doped Cz Si	250–350	10^{-13}	0.27	32
(g)	^{63}Ni: autoradiography in KDB-7.5 Si	597–797 197–597	2.3×10^{-3} 3.4×10^{-12}	1.7 0.13	33 33

[a] AES stands for Auger electron spectroscopy.

[b] D at 800 °C.

V, Cr, Mn, and Fe,[3] and in all probability Ni^+ atoms are electrically inactive. Although the role of positively charged Ni atoms is not determined, it is generally accepted that they reside and migrate as interstitials in the Si lattice. If Ni were to occupy and to diffuse via the substitutional sites, firstly it would be electrically active and secondly its diffusion would show a large activation energy of the order of 3–4 eV similar to P or (Refs. 23 and 24) B in Si. There are, obviously, contrary to the observations. For many impurities in Si such as Au, a "kick-out" mechanism[20] has been proposed which requires intervention—production and annihilation—of Si self-interstitials. In the case of Ni, however, such a cooperative reaction as we discussed is not considered possible in view of the fast diffusion at all temperatures.[20] The growth mechanism of Ni_2Si, NiSi, and $NiSi_2$ silicides on Si(100) and Si(111) surfaces have been studied extensively as reported in the literature.[25] To determine the migrating species in the Ni silicides, markers[26] and radioactive ^{31}Si (Ref. 27) and ^{63}Ni (Ref. 28) tracers have been employed. Ni appeared to be the dominant moving species diffusing through the silicides and into Si at the Ni-silicide/Si interface.[29] The rapid growth kinetics of the Ni silicides can be reconciled only with the interstitial diffusion of Ni in Si, at least in the close vicinity of the interface.

B. Investigations yielding low diffusivities of Ni in Si

We now turn our attention to those investigations listed in Table I [(e)–(g)] and shown in Fig. 3 which result in much smaller diffusion coefficients for Ni atoms in Si. Since a variety of preexponential factors and activation energies are encountered in these investigations, we will discuss them individually.

As seen in Table I [investigation (e)] the diffusion study of ^{63}Ni in single crystal Si by Bonzel[30] leads to $D_0 = 1000$ cm^2/sec and $Q = 4.24$ eV. His technique involves electroplating of the ^{63}Ni tracer onto the Si surface and monitoring the attenuation of the ^{63}Ni radioactivity during the diffusion process. The technique is susceptible to the growth of oxides at the surface. The influence of ambients on the growth of surface compounds and on diffusion is discussed by Chang.[31] Briefly, the kinetics are described by the "sinking" of the interface as the substrate material migrates to the surface forming a compound there, for instance silicon oxide in the present case. This situation can be construed from the high activation energy of the order of 4 eV measured in this investigation, which is not too different from the value for self-diffusion in Si.[20] Therefore these values are suspect.

The low-temperature-diffusion investigation of Ni in (100) Si by Berning *et al.*[32] using Auger-electron spectroscopy (AES) leads to unrealistic values for the preexponential factor ($\sim 10^{-13}$ cm^2/sec) as well as for the activation energy (0.27 eV) and cannot be attributed to a reasonable mechanism of diffusion in solids. The results of the investigation by Uskov *et al.*[33] are not linear in the Arrhenius plot as may be seen in Fig. 3. They used two exponentials to account for high- and low-temperature processes. The parameters for the high-temperature process are too large and those for the low-temperature process are too small for any reasonable diffusion mechanism. Therefore values from these investigations are considered inadmissible. We attempted to extract diffusion coefficients from our profiles and the results were similar to those of Uskov *et al.*[33] However, this procedure must be rejected for the reasons described earlier.

IV. SUMMARY

The published coefficients for Ni diffusion in Si show a scatter over ten orders of magnitude. Our ^{63}Ni diffusion measurements in P-doped Cz Si employing the percolation and autoradiographic techniques have narrowed this uncertainty to within one order of magnitude over the 220–540 °C temperature range. A critique of the diffusion data in the literature reveals the experimental and base-material-related difficulties many of the investigators might have faced. As a consequence, the reliable data yield a high diffusion coefficient even in the low-temperature regime and a low solubility of Ni in Si. These data are indicative of an interstitial mechanism of diffusion for Ni atoms in the Si lattice. The "kick-out" mechanism is ruled out on the basis of the observed diffusion profiles and parameters.

ACKNOWLEDGMENTS

The authors would like to thank Dr. B. S. Berry and R. D. Thompson for helpful discussions.

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