Simulation of chemical composition nitrogen-argon-aluminum plasma

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Abstract. Plasma-chemical model of magnetron discharge is described. The model allows you to predict the densities of the main plasma components. As a result of numerical modeling, it was found that dissociation of \( \text{N}_2 \) ranges from 10% to 30% with an increase in plasma power, the degree of dissociation increases. The addition of \( \text{N}_2 \) to the plasma leads to a decrease in ion density due to energy consumption for non-ionizing molecular collisions. It can be assumed that aluminum nitride is deposited on the surface of a substrate with a high flow of metal ions in combination with a stream of neutral molecules \( \text{N}_2 \) with a low adhesion coefficient and a relatively small flow of nitrogen ions.

1. Introduction
The optical, electrical, dielectric and acoustic properties of aluminum nitride (AlN) have recently stimulated a great deal of interest. Films are suitable as insulating and passivation layers in semiconductor devices due to chemical heat resistance, as well as high resistivity. In addition, aluminum nitride films are used in the design of devices for selecting and generating signals in the microwave range on bulk acoustic waves (BAW) due to a sufficiently high value of the electromechanical coupling constant (up to 0.07), which provides piezoelectric properties of the film, and a high velocity of the longitudinal acoustic wave (up to 11000 m/s). Aluminum nitride films can be prepared by chemical vapor deposition (CVD), molecular beam epitaxy, etc [1]. In these methods, the deposition temperature of the substrate was sufficiently high. For example, Morita et al. [2] obtained an epitaxial growth of AlN on silicon using an organometallic CVD system at a substrate temperature of 1260 °C. In alternative atomic layer deposition system, the films were coated by the reaction of aluminum tribromide and ammonia in a temperature range of 600-900 °C. In order to provide a single processing cycle in the production of semiconductor devices, there is a need to develop methods for depositing AlN films at a temperature of less than 400 °C. Low temperature deposition reduces thermal voltages in the insulator-semiconductor interface and also allows the manufacture of devices that require a metal electrode under the film. Magnetron (RF or DC) sputtering techniques [3, 4] are commonly used to deposit films at relatively low temperatures. Magnetron Sputtering localizes the plasma near the surface of the target cathode, increasing the sputtering efficiency.

To better understand the processes of forming aluminum nitride films using gas-discharge plasma, a model is presented (averaged by volume) describing the physicochemical processes in the Al/Ar/N\(_2\)-
plasma of the glow discharge. Modeling of plasma Al/Ar/N₂ begins with the selection of key reactions in discharge and solving mass conservation (particle balance) and energy conservation (power balance) equations. For full gas pressure and flow rate, Al vapor phase density and absorbed power, the Al/Ar/N₂ plasma model can predict electron temperature and concentrations of primary plasma components, such as N, N⁺, N₂, N₂⁺, Ar⁺, Ar, e⁻ and Al⁺.

2. Basic model provisions
- given particle densities averaged by volume;
- one equivalent excited state of N₂ in plasma is considered. Generation of N₂⁺ is determined from the general section of electronic excitation for N₂;
- due to low probability of reaction at pressures of interest (10⁻⁴ to 10⁻⁵ mTorr), collisions involving three particles are not included in the model;
- temperatures of neutral and ionic species are taken to be amount to the gas temperature N₂;
- electronic dissociation of nitrogen ion N₂⁺ is not taken into account, since the molecular density of ions is relatively small compared to the density N₂;
- sprayed Al atoms are completely thermalized when they collide with the gas mixture Ar/N₂;
- due to lack of data on the ionization section of the excited state of aluminum (Al⁺+e⁻→Al⁺+2e⁻), it is assumed that Al⁺ generated only by a single-stage electronic impact ionization process Al⁺+e⁻→Al⁺+2e⁻ and by Penning ionization Al⁺+Ar→Al⁺+Ar+e⁻;
- aluminum nitride is mainly formed on the substrate during reactive sputtering of aluminum (Al) in Ar/N₂ plasma. Therefore, reactions Al_gas+N_gas→AlN_gas in the gas phase can be neglected.

3. Energy balance
For the energy balance, it is necessary to take into account the speed, pressure and density of aluminum atoms in the vapor phase. This model for plasma Al/Ar/N₂ corresponds to the general Lee and Lieberman approach [5].

The addition of N₂ to the system results in a much more chemical complex environment, both in the gas phase and on the surface. The strategy is to first develop the generation rate for each of the types of plasma components. Various loss mechanisms will then be described. When comparing generation rates and losses, temperature of electrons and density of each plasma component can be self-consistently determined.

4. Particle generation
In this plasma model, the main species are the basic states of Al, Ar and N₂, excited atoms N₂⁺ and Ar⁺, N, electrons, ions Al⁺, N₂⁺, N⁺ and Ar⁺. For simplicity, we combine all excited argon or nitrogen states into one state, called Ar⁺ and N₂⁺, respectively. A simplified set of volumetric reactions and corresponding velocity constants is shown in table 1. Reactions associated with N₂ include ionization, dissociation, recharging, and excitation. Argon-related reactions included excitation and electron-impact ionization. Charge exchange reactions between Ar⁺ and N₂ in the table 1. By numerical integration were calculated rates constants of electron-induced reactions:

\[ K_j(T_e) = \langle \nu \sigma_j \rangle = \int_{E_t}^{\infty} \nu \sigma(E)f(T_e,E) dE, \]  \hspace{1cm} (1)

where \( \nu \) is velocity of the electron, \( \sigma(E) \) is impact cross section of the electron, \( f(T_e,E) \) is the normalized Maxwell’s electron distribution function \( E_t \) is the reaction threshold energy. To achieve numerical convergence of the model, the calculated velocity constants correspond to the Arrhenius formula:

\[ K(T_e) = K_0(T_e) e^{\frac{-E_0}{T_e}}, \]  \hspace{1cm} (2)
where $K_0$, $E_0$ and $C$ are the constants given in table 1.

5. Particle loss mechanisms

Working pressure of processes at precipitation of aluminum nitride in magnetron discharge is from 10 to 40 mTorr. In this range, the probability of electron-ion recombination in the pressure discharge volume is negligible. Thus, the main mechanism of ion loss is their diffusion to walls of the chamber. The rate of diffusion loss is determined by the lifetime of the ions:

$$\tau_i = \frac{A^2}{D_0}$$  \hspace{1cm} (3)

where $A$ is the chamber characteristic diffusion length, $D_0 = \frac{k_B e \mu_i}{e}$ is coefficient of the ambipolar diffusion, $k_B$ is the Boltzmann’s constant, $T_e$ is electron’s temperature, $\mu_i$ is ion’s mobility, $e$ is electron charge.

| Reaction                                      | Rate constant                                      | Reference |
|-----------------------------------------------|----------------------------------------------------|-----------|
| $N + e^- \rightarrow N^+ + 2e^-$              | $K_1 = 3.8 \times 10^{-9}(T_e)^{0.92}\exp(-12.1/T_e)\text{cm}^3\text{s}^{-1}$ | [6]       |
| $N_2 + e^- \rightarrow 2N + 2e^-$              | $K_2 = 2.1 \times 10^{-9}(T_e)^{1.13}\exp(-14.4/T_e)\text{cm}^3\text{s}^{-1}$ | [7]       |
| $N_2 + e^- \rightarrow N^+_2 + e^-$            | $K_3 = 6.2 \times 10^{-9}(T_e)^{0.81}\exp(-12.8/T_e)\text{cm}^3\text{s}^{-1}$ | [8]       |
| $N^+ + N_2 \rightarrow N^+_2 + N$              | $K_5 = 2.0 \times 10^{-11}\text{cm}^3\text{s}^{-1}$ | [9]       |
| $N + N^+_2 \rightarrow N^+ + N_2$              | $K_6 = 1.0 \times 10^{-11}\text{cm}^3\text{s}^{-1}$ | [10]      |
| $N^+_2 + N \rightarrow N_2 + N_2$              | $K_7 = 4.0 \times 10^{-11}\text{cm}^3\text{s}^{-1}$ | [11]      |
| $N^+_2 + N_2 \rightarrow 2N_2$                  | $K_8 = 3.5 \times 10^{-12}\text{cm}^3\text{s}^{-1}$ | [12]      |
| $N_2 + e^- \rightarrow N^+_2 + e^-$            | $K_9 = 5.8 \times 10^{-9}\exp(-7.57/T_e)\text{cm}^3\text{s}^{-1}$ | [13]      |
| $N_2^+ \rightarrow N_2 + hv$                   | $\tau_{hv} = 2.3 \times 10^{-4}\text{s}$         | [14]      |
| $W(\text{wall}) = 0.5 \cdot N_2^+(g)$         | $\tau_{N_2^+}^{-1} = D_N/A_{N_2^+}^2\text{s}^{-1}$ | [15]      |
| $Ar^+ + N_2 \rightarrow N_2^+ + Ar$            | $K_{10} = 1.2 \times 10^{-11}\text{cm}^3\text{s}^{-1}$ | [16]      |
| $Ar + N_2 \rightarrow Ar^+ + e^-$              | $K_{11} = 2.2 \times 10^{-8}\exp(-12.4/T_e)\text{cm}^3\text{s}^{-1}$ | [17]      |
| $Ar^+ + e^- \rightarrow Ar^+ + 2e^-$           | $K_{12} = 2.1 \times 10^{-7}\exp(-5.3/T_e)\text{cm}^3\text{s}^{-1}$ | [17]      |
| $Ar + e^- \rightarrow Ar^+ + 2e^-$             | $K_{13} = 1.2 \times 10^{-7}\exp(-18.68/T_e)\text{cm}^3\text{s}^{-1}$ | [18]      |
| $Al + e^- \rightarrow Al^+ + e^-$              | $K_{14} = 2.3 \times 10^{-7}\exp(-7.25/T_e)\text{cm}^3\text{s}^{-1}$ | [19]      |
| $Al + Ar^+ \rightarrow Al^+ + Ar + e^-$        | $K_{15} = \sigma_{Al} \cdot \nu_{th,Al} \text{cm}^3\text{s}^{-1}$ | [19]      |

6. Particle balance

The rates of generation and loss for each particle can be determined based on the reactions shown in table 1. By equating the generation rates with the loss rates, a group of equations is made for stationary particle densities. This method is essentially conservation of mass and is often called to as particle balance. As a case, for atomic nitrogen, consider the first row in table 2. In this reaction set, there are two processes that it is formed $N$: first one is dissociation of $N_2$ and charge exchange reaction between $N^+$ and $N_2$. The generation rate $N$ is $2K_i[e][N_2] + K_d[N^+] [N_2]$, where $[e]$, $[N_2]$ and $[N^+]$ are electron, $N_2$ and $N^+$ densities, respectively, $K_i$ and $K_d$ are the dissociation and charge transfer rate constants, respectively (see table 1). The loss conditions for $N$ are ionization of $N$, charge transfer between $N$ and $N_2^+$ and diffusion losses. The loss coefficient $N$ is written as $K_i[e][N] + K_d[N] [N_2] + ([N] / \tau_N)$, where $\tau_N$ is the atomic nitrogen’s diffusion lifetime, $K_i$ is rate constant of $N$ ionization, and $K_d$ is the rate constant for charge exchange reaction. Similarly, particle balance equations are obtained for each of the plasma species.
Table 2. Particle balance equations for Al/Ar/N₂ plasma.

| Species | Equation |
|---------|----------|
| N⁻² | \(2K_3[e][N_2] + K_6[N^+][N_2] = K_4[e][N] + K_8[N^+][N] + \tau_{N^+}^{-1}[N] \) |
| N⁺ | \(K_1[e][N] + K_6[N][N^+] = K_N[N^+][N] + \tau_{N^+}^{-1}[N^+] \) |
| N₂ | \(K_0[e][N_2] = K_7[N][N_2^+] + K_9[N_2^+][N_2] + \tau_{N_2^+}^{-1}[N_2^+] + \tau_{N_2^+}^{-1}[N^+] \) |
| N₂⁺ | \(K_2[e][N_2] + K_5[N^+][N_2] + K_{10}[Ar^+][N_2] = K_6[N][N^+][N_2] \) |
| Ar⁺ | \(K_{12}[e][Ar^+] + K_{13}[e][Ar] = K_{10}[Ar^+][N_2] + \tau_{Ar^+}^{-1}[Ar^+] \) |
| Ar⁺⁺ | \(K_{11}[e][Ar] = K_{12}[e][Ar^+] + K_{15}[Al][Ar^+] + \tau_{m}^{-1}[Ar^+] + \tau_{Ar^+}^{-1}[Ar^+] \) |
| Al⁺⁺ | \(K_{14}[e][Al] + K_{15}[Ar^+][Al] = \frac{[Al^+]}{\tau_{Al^+}} \) |

7. Energy balance

If the electron density is known, the equations given in table 2 can be solved. In this part, the density of electrons in the plasma is calculated by matching the power supplied to the discharge with the loss energy in plasma. In plasma, there are three energy loss channels: the loss of electron energy as a result of all electron-neutral plasma collision processes, the loss of ion energy on the reactor wall, and the loss of energy on the walls.

Electron energy losses in all electron-neutral collisions are given as:

\[
P_{ev} = e n_e V \sum_{i=1}^{N_e} \nu_{iz,i} \varepsilon_{L,i} \nu_{L,i} \quad (4)
\]

where \(e\) is elementary charge, \(n_e\) is electrons density averaged by the volume, \(V\) is volume of the reactor, \(\nu_{iz,i}\) is ionization frequency for generation of \(i-th\) ion, \(\varepsilon_{L,i}\) is the overall energy loss in a collision on an electron-ion pair generated for the \(i-th\) ion, \(a\) is amount of positive ions generated in plasma. Energy losses per electron-ion pair resulting from electron-neutral collision processes \(\varepsilon_{L,i}\) can be described as follows:

\[
\nu_{iz,i} \varepsilon_{L,i} = \sum_{j=1}^{N_{s,i}} \left( \nu_{iz,i} \varepsilon_{iz,i} + \nu_{ex,i} \varepsilon_{ex,i} + \nu_{diss,j} \varepsilon_{diss,j} + \nu_{el,j} \frac{3m}{M_j} T_e \right), \quad (5)
\]

where \(N_{s,i}\) is amount of neutral species that create for the production of the \(i-th\) ion, for example, for \(Ar^+\), \(N_{s,i}=2\) (\(Ar\) and \(Ar^+\)), \(\nu_{iz,i}\) is the is the ionization frequency to form the \(i-th\) ion from neutral particles \(j\), \(\varepsilon_{iz,i}\) is the ionization threshold energy for \(i-th\) ion from neutral species \(j\), \(\nu_{ex,i}\) is frequency of excitation the \(j-th\) neutral, \(\varepsilon_{ex,i}\) is the threshold energy of excitation \(j-th\) neutral, \(\nu_{diss,j}\) is the dissociation frequency through electron impact, \(\varepsilon_{diss,j}\) is threshold energy of dissociation the \(j-th\) neutral, \(\nu_{el,j}\) is the frequency of elastic impact between the electron and the \(j-th\) neutral, \(3m/M_j\) is mean energy of loss per electron with \(m\) mass through elastic scattering on the \(j-th\) neutral \(M_j\) mass. Each of the collision frequencies of electrons is determined by \(v=Kn\), where \(K\) is the corresponding rate constant given in table 1, \(n\) is density of the heavy particle collision. Table 3 lists threshold energies of collisions to account in this model. Since for excited argon is no single ionization threshold energy, it’s can be estimate as the difference between the direct ionization energy and the excitation threshold energy.

It should be noted that collision reactions in molecular nitrogen result in significantly higher energy consumptions per ion than in atomic argon.
Table 3. Threshold energy of collisions in argon/nitrogen plasma.

| Process                                      | Threshold energy, eV | Reference |
|----------------------------------------------|----------------------|-----------|
| \( N + e^- \rightarrow N^+ + 2e^- \)       | 14.54                | [20]      |
| \( N_2 + e^- \rightarrow N_2^+ + 2e^- \)   | 15.6                 | [21]      |
| \( N_2 + e^- \rightarrow 2N + e^- \)       | 9.76                 | [22]      |
| \( N_2 + e^- \rightarrow N_2(A) + e^- \)   | 6.17                 | [23]      |
| \( Ar + e^- \rightarrow Ar^+ + 2e^- \)     | 15.76                | [24]      |
| \( Ar^* + e^- \rightarrow Ar^* + e^- \)    | 4.2                  | [24]      |
| \( Ar + e^- \rightarrow Ar^* + e^- \)      | 11.55                | [24]      |

Losses of ionic energy on the walls are determined by the flow of ions to the walls and the potential of the plasma sheath:

\[
P_{iw,i} = en_{s,i}u_iA\epsilon_{iw,i}
\]  

(6)

where \( n_{s,i} \) is the density \( i \)-th ion at edge of the sheath, \( u_i \) is the velocity of \( i \)-th ion, \( A \) is chamber's walls surface area, \( \epsilon_{iw,i} \) is kinetic energy expense of ion to the wall. Usually, ions in plasma receives directed energy of \( T_e/2 \) in the presheath region such that the velocity of the ion entering the sheath is equal to the velocity, \( u_M = \sqrt{eT_e/M} \). The additional ion energy is determined by the sheath voltage \( V_s \), which is

\[
V_s = \frac{T_e}{2} \ln \left( \frac{M}{2\pi m} \right).
\]  

(7)

The energy loss of electrons on the wall is:

\[
P_{ew} = en_{es}u_MA\epsilon_{ew},
\]  

(8)

where \( n_{es} \) is the electron density at edge of sheath, \( \epsilon_{ew} \) is kinetic energy of the electron shaded to wall (\( \sim 2T_e \) per electron that reaches of wall).

Figure 1. Energy losses per electron-ion pair generation in \( N_2/Ar \) plasma.
Electron density at the edge of the layer is associated with ion density by quasi-neutrality condition:

\[ n_{es} = \sum_{i=1}^{r} n_{is} \]  \hspace{0.5cm} (9)

In order to calculate power losses on the walls, the ratio is determined between ion concentration at the axial sheath edge \( n_{isl} \) and their density \( n_i \) at center of the plasma:

\[ h_L \equiv \frac{n_{isl}}{n_i} \approx \frac{0.86}{\sqrt{3+\frac{r}{2}x_i}} \]  \hspace{0.5cm} (10)

this equation is used at the edge of the radial layer. In both equations (5) and (10), the mean free ion path is approximated for \( \text{Ar}^+ \), the most common type of ion.

As a result, three power loss channels add up, and then the balance of power in the plasma can be represented as:

\[ P_{abs} = P_{ev} + \sum_{i=1}^{r} P_{1W,i} + P_{ew}, \]  \hspace{0.5cm} (11)

where \( P_{abs} \) is power absorbed by the plasma, \( r \) is amount positive ions generated in plasma. By self-consistent solving of equation (11), the particle balance equation in table 3, and the quasi-neutrality condition in equation (9), it is possible to calculate the electron temperature, electron density, and average density of all other plasma species.

The total pressure is considered as the pressure before discharge is ignited. The total particle density before plasma ignition is defined as:

\[ n_{tot} = \frac{p}{k_B T_{gas}} \]  \hspace{0.5cm} (12)

where \( p \) is pressure in vacuum chamber, \( T_{gas} \) is the gas temperature. Initial density of the \( N_2 \) is determined by:

\[ n_{N_2} = \frac{F_{N_2}}{F_{N_2} + F_{Ar}} \cdot n_{tot}, \]  \hspace{0.5cm} (13)

where \( F_{N_2} \) and \( F_{Ar} \) are flow rates of molecular nitrogen and argon, respectively. The actual \( N_2 \) density after plasma formation is determined from the mass conservation. All input parameter values are experimental data.

Pressures varied from 15 to 30 mTorr, argon flow rate was kept constant at 50 sccm, and nitrogen flow rate was changed from 0 to 10 sccm. As a rule, the discharge power is from 500 to 1500 watts.

**Results and discussion**

Figure 2 shows the degree of nitrogen dissociation depending on the total gas pressure and power. Dissociation increases in proportion to plasma power, is about 10% -15% at low discharge power (750 W) and rises up to 30% at 1.5 kW power.

The model contains the parameter of loss of a wall for nitrogen atoms \( \gamma_N \) which can be defined by comparison of the simulated results for density of atoms of nitrogen with experimental mass and spectrometer measurements of \( N/N_2 \), this parameter is equal in our case to one.
Figure 2. Degree of nitrogen dissociation depending on pressure and power.

Figure 3 shows the densities of all simulated plasma components depending on the pressure at fixed flow rates of 5 sccm $N_2$, 50 sccm $Ar$, and $[Al] = 0$. The starting gases dominate the composition of the plasma, but the density of atomic nitrogen is also significant. The total ion density increases with increasing pressure. Primary types of ions are $Ar^+$ and $N_2^+$ at the same time nitrogen ions, less plentiful as argon's ions, and density of $N^+$ does not exceed of $10^9 \text{ cm}^{-3}$. From figure 3 seen that the proportion of ionization of $N_2$ and $N$ about 0.1%. This ionization is much smaller than the mass fraction of the ionization of metal atoms in plasma systems, which is usually about 10-30%. Thus, the flow of metal entering the substrate is strongly ionized and can be directed by the potential of the plasma sheath, but the flow of nitrogen is predominantly neutral and uncontrollable by the sheath potential.

Figure 3. Density of plasma $Ar/N_2$ ions depending on pressure at discharge power of 1 kW.

The proportion of ionized metal flow is calculated by the formula $\Gamma_{Al^+}/(\Gamma_{Al^+}+\Gamma_{Al})$. When sputtered $Al$ is added to the discharge, it is mainly thermalized within a few centimeters when it collides with background gas $Ar/N_2$. At low pressure or short throw distance, some $Al$ atoms can pass through the discharge without achieving thermal equilibrium with the plasma. However, at the
pressures used, the calculation of the flow $A_l$ is relatively simple, since aluminum quickly reaches thermal equilibrium with gas, the flow of thermalized atoms $A_l$ to the plate from the discharge is:

$$
\Gamma_{A_l} = \frac{1}{4} v_{th,A_l}[Al] = \frac{1}{4} \sqrt{\frac{3k_B T_{gas}}{\pi M_{A_l}}} [Al],
$$

(14)

where $v_{th,A_l}$ is the $A_l$ mean thermal velocity. The $A_l$ ion flux on the wafer is calculated by:

$$
\Gamma_{A_l^+} = 0.6[A_l^+] \frac{k_B T_e}{M_{A_l^+}}
$$

(15)

The general density of the sputtered $A_l$ depends on power supplied, in our case density will be about $10^{11} \text{ cm}^{-3}$ for calculation in figure 4, (i.e., $[Al]_0 = [Al] + [A_l^+] = 10^{11} \text{ cm}^{-3}$). The density of $A_l$ is typical for such discharges, it has been found that the proportion of ionized $A_l$ is practically independent of $A_l$ volume incoming to the plasma for densities of $[Al] < 10^{12} \text{ cm}^{-3}$. For moderate plasma power (less 1kW), the model shows that electron temperature is about 3-4 eV, and the fraction of ionized aluminum flow reaching the surface of a substrate is $\Gamma_{A_l^+}/(\Gamma_{A_l}+\Gamma_{A_l^+}) \sim 0.8$.

![Figure 4. Fraction of aluminum ion fraction depending on electron temperature.](image)

The deposition rate can be calculated by the formulae (14, 15) if the applied film is $AlN$ almost stoichiometric as observed in the nitride mode. Under this condition, there is nitrogen plenty in the plasma and the deposition is rate limited by the flux of aluminum atoms and ions.

Thereby, the deposition rate is:

$$
R_{dep} = \frac{S(\Gamma_{A_l}+\Gamma_{A_l^+})}{n_{AlN}},
$$

where $S$ is the sticking coefficient for $A_l$ on $AlN$ ($\sim 1$), and $n_{AlN}$ is the number density of $AlN$ within the film [24].

**Conclusion**

The dissociation of $N_2$ is in the variety of 10% - 30% and rises with increasing plasma power. The addition of $N_2$ to the plasma leads to a decrease in ion density due to the energy that is divided into non-ionizing molecular collisions. As a rule, the total ion density is in variety of $2 - 6 \times 10^{11} \text{ cm}^{-3}$. 
Nitrogen gas has a much lower ionization fraction than Al due to a significantly higher ionization potential. These conclusions suggest that aluminum nitride is deposited on the surface of a substrate with a high flow of metal ions in combination with a stream of neutral molecules $N_2$ with a low adhesion coefficient and a relatively small flow of nitrogen ions.

As a result of the studies carried out, it was possible to develop a model that adequately describes gas discharge plasma when depositing thin films of aluminum nitride. It is necessary to directly compare the results with experimental data in order to unequivocally assert the correctness of this approach in the modeling of nitrogen-argon-aluminum plasma.

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