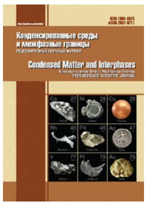

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A model of interdiffusion occurring during the formation of thin metal films on single-crystal silicon under conditions of limited solubility of the components

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Abstract

Thin metal films are used in semiconductor and microelectronic devices to form ohmic and non-ohmic contacts to single-crystal silicon. A common feature of the used Me–Si systems is the low mutual solubility of elements and the polycrystalline nature of metal films. Solid-phase interactions during the deposition of metals on single-crystal silicon and the subsequent vacuum annealing results in the redistribution of the elements near the Me/Si interface. An important task facing the material science of solid-state electronics is to develop a mechanism of solid-phase interaction of metal thin films and single-crystal silicon. The aim of our study – was to develop a quantitative model of interdiffusion in the Me–Si system under conditions of limited solubility of the components.

The article suggests a mechanism of formation of Me–Si systems based on the diffusion and segregation of silicon near the intergrain boundaries of the metal and the limited formation of complexes during the diffusion-induced penetration of metal into silicon. The article suggests a model of reactive interdiffusion in thin metal film – single-crystal silicon systems under conditions of limited solubility of the components. Mathematical modelling was used to study the interaction of magnetron-sputtered metals Ti, W, and Nb with single-crystal silicon during isothermal vacuum annealing. The numerical analysis of experimental distributions of concentrations of Me and Si obtained by Rutherford backscattering spectroscopy allowed us to determine their individual diffusion coefficients in Me-Si systems.

The model can be used for empirical studies of the redistribution of the elements of two-layer systems with limited solubility, as well as to forecast the technological conditions for the production of electronic devices.

Keywords: Modelling, Reactive interdiffusion, Limited solubility, Metal thin films, Single-crystal silicon.

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1. Introduction

Thin films of metals such as Al, Pt, Nb, Au, Ag, Co, Pd, Ni, Ti, and W [1] are used in semiconductor and microelectronic devices to obtain ohmic and non-ohmic contacts to silicon. The contacts are typically constructed by depositing a metal on silicon followed by annealing in an inert medium. The interaction between the metal and the semiconductor is considered to have the following effect: the presence of the metal in the crystal lattice of silicon results in the weakening and breaking of covalent Si-Si bonds and the formation of mobile silicon atoms [2]. The flow of silicon atoms is directed towards the metal-silicon interface, while the metal atoms flow in the opposite direction. This results in the interdiffusion and redistribution of both metal in silicon and silicon in the metal film. The difference in the diffusion coefficients of the atoms moving towards each other results in the Kirkendall effect [3], which comes in the form of the motion of the interface in the diffusion couple.

Physicochemical interaction between metals and silicon results in the formation of eutectic systems. When the electronegativity of the diffusing metal atoms is significantly different from that of Si, a solid-phase reaction of the formation of silicide can take place [1, 4]. It is assumed that silicide phases with various concentrations of silicon and metal are formed layer-by-layer on the Me/Si interface [5].

A common feature of the used Me-Si systems is that after its deposition on silicon and during the following annealing in an inert medium, the metal remains in a polycrystalline state. The heat treatment facilitates the faster movement of grain boundaries in the metal and faster diffusion of Si in the metal film. However, the existing understanding of the interdiffusion in the Me-Si system does not take into account the role of grain boundaries in the process of diffusion-controlled growth of silicide phases and the formation of solid solutions over a wide range of temperatures.

Another feature of the used Me-Si systems is the limited solubility of silicon in the crystallites of metal and limited solubility of metal in single-crystal silicon. The existing literature does not provide enough details regarding the diffusion mechanism of the used metals in silicon and diffusion of silicon in metal.

To control the redistribution of the components near the metal-silicon interface and obtain the electrical and technological parameters of the formed structures required for the semiconductor and microelectronic devices, it is necessary to develop a mechanism for the solid-phase interaction of thin metal films and single-crystal silicon. Therefore, an important task facing the material science of solid-state electronics is to simulate the processes of interdiffusion, segregation, and solid-phase chemical reactions in the Me-Si system.

The purpose of our study was to develop a model of interdiffusion occurring during the formation of the thin metal film – a single-crystal silicon system under conditions of limited solubility of the components.

2. Description of the model

Let's assume that when a metal interacts with single-crystal silicon, its crystal lattice partially disintegrates. Some of the silicon atoms from the crystal lattice become free and transfer to a free mobile state. In the single-crystal Si, they move as self-interstitial atoms, while in the metal film they move – in the intergrain space.

Silicon has low solubility in metal crystallites. Therefore, regarding the dissolution of silicon in the metal film, we shall assume that silicon does not diffuse and dissolve in the crystallites of metal. However, under conditions of low solubility in crystallites, deep diffusion of silicon may occur along the grain boundaries together with the solubility in the intergrain space containing a large concentration of defects. As a result, the solubility limit of silicon in the intergrain space may be significantly higher than that in the crystallite volume.

Grain boundaries contain unsaturated coordinate bonds appearing on the surface of the metal grains and serve as trap centres for silicon, characterising the presence of vacant positions for silicon trapping. We assume that the mechanism of silicon trapping by metal is accompanied by a release of free metal (silicon replaces metal in the traps). Then, the formation of a solid solution of silicon in metal can be described as a diffusion penetration of mobile silicon from the single-crystal phase to the intergrain space of the metal film followed by a partial segregation trapping by vacant trap centres. As a result, Me and Si atoms moving in the film become immobilised on the Me grain boundaries, losing their mobility.

This stage of the process is carried out by the mechanism of physical adsorption without chemical interactions resulting in the formation of compounds (silicides). The stage is topochemical and takes place in the Me film volume as a result of diffusion penetration of silicon. It is localised on the grain boundaries where the energy loss induced by bond deformation is minimal and there is some free volume facilitating the reorientation of the reacting particles. The trapping of silicon proceeds until there are no vacant traps. Along with the process of silicon trapping, the solution becomes saturated with dissolved silicon. In terms of numerical value, the concentration of traps corresponds to the solubility limit of silicon in the intergrain space of a polycrystalline metal and constitutes fraction *r* of the total concentration of the metal.

Regarding the process of dissolution of metal in the single-crystal silicon phase, let's assume that along with the diffusion penetration of metal into silicon, the decomposition of the solid solution of metal in silicon takes place resulting in the formation of stationary multiparticle complexes containing intrinsic point defects in silicon and metal atoms. This process limits the solubility of metal in single-crystal silicon.

A large number of metals can produce silicides with various concentrations of components, when interacting with silicon. With regard to the suggested mechanism, this ability can be interpreted as follows. The intergrain space of the metal film has an increased concentration of defects and therefore enough free volume for the potential formation of silicides whose molar volumes are different from that of the metal. During the later stages of the process and with an increased annealing temperature, a chemical absorption stage with the formation of metal enriched silicides can occur near the Me/Si interface, on the side of the metal film and in its depth. On the silicon side of the Me/Si interface, silicon enriched silicides can be formed based on the obtained complexes. The required free volume will be provided for by the intrinsic point defects in silicon. In this case, silicides do not grow layer-by-layer on the Me/Si interface. Rather, they are formed inside an extended reaction zone next to it compatible with the initial Me film. The large extent of this zone indicates a relatively slow solid-phase reaction as compared to the rate of diffusion of mobile Si and metal atoms.

[6] suggested a convenient (from the point of view of numerical analysis) mathematical description of the interdiffusion process for a binary system with unlimited solubility. It does not take into account the chemical transformations that may occur during the interdiffusion and is based on the assumption that the molar volume of the system is invariable, regardless of the changes in its composition. In [6], the process is described by means of a boundary value problem for two diffusion equations which contain an effective interdiffusion coefficient which is a linear combination of individual diffusion coefficients of all the components.

The theory suggested in [6] is applied to the formation and growth of interface phases in the diffusion zone [7-10].

[11] for the first time developed the theory suggested in [6] as applied to bulk reactions of the formation of metal silicides during the interaction between the film of the silicide-forming Ni and SiC. However, the model suggested in [11] cannot be used to describe the redistribution of the components inside an extended reaction zone. We believe that this can be down to the fact that the model does not account for the changes in the molar volume of the system during the solid-phase reaction of the formation of silicides.

[12–14] used the mathematical description from [6] in a quantitative model which developed the theory suggested in [6] as applied to the reactive interdiffusion in two-layer metal-metal oxide systems with limited solubility of the components. This model can be used to describe the formation of complex polycrystalline Co–TiO_2 and Fe–TiO_2 thin-film systems which form not by layer-by-layer growth at the metal-oxide interface, but throughout the film thickness of TiO_2.

[15] developed the theory suggested in [6] and introduced a model of reactive interdiffusion in nonstoichiometric polycrystalline oxide film systems with limited solubility under vacuum annealing conditions. The model was used to describe the formation of the phases of complex

oxides distributed along the depth of the system of two nonstoichiometric polycrystalline oxides of titanium and cobalt [16].

The application of the model suggested in [6] to the metal film - single-crystal silicon system with limited solubility of the components has not been studied yet.

In our study, we considered the following components of the system.

– Stationary silicon atoms A which constitute the lattice of single-crystal silicon.

– Mobile silicon atoms B formed as a result of interaction between the metal and silicon.

– Vacant trap centres Ct for mobile silicon in the intergrain space of the metal.

– Silicon atoms Bt trapped in the intergrain space of the metal.

– Mobile (free) metal atoms C.

– Stationary complexes C_p in silicon containing metal and silicon atoms.

The total concentration of silicon was comprised of the following elements:

– concentration of silicon C_A at the points of the silicon crystal lattice A;

– concentration C_B of mobile silicon B;

– concentration C_{Bt} of silicon trapped by Bt traps in the intergrain space of the metal; and

– silicon concentration C_{Cp} in metal-silicon complexes C_p:

$C_A + C_B + C_{Bt} + C_{Cp}$.

The total concentration of metal was comprised of the following elements:

– concentration of mobile (free) metal C;

– metal concentration in the vacant trap centres for mobile silicon in the intergrain space of the metal C_{Bt};

– metal concentration C_{Cp} in metal-silicon complexes C_p:

$C_C + C_{Bt} + C_{Cp}$.

The elements of the system capable of diffusion-induced migration are mobile metal atoms C and mobile silicon atoms B released in the presence of the metal from the crystal lattice of silicon. If the beginning of the reference system is the outer border of the Me film, then, with the ratio of individual diffusion coefficients being ($D_C >> D_B$), the Me-Si grain boundary will move towards the surface of the film system as a result of the Kirkendall effect [3]. The elements not capable of diffusion-induced migration will serve as inert markers in the Smigelskas and Kirkendall experiment [3].

In our study, the qualitative nature of the process determines that the molar volume of the system is invariable. Therefore, we assume that the diffusion of Me and Si in the Me-Si system can also be described by means of the mathematical formalism suggested in [6]. Let’s determine the effective interdiffusion coefficient for the simulated system as

$$D^* = \frac{D_B \cdot C_C + D_C \cdot C_B}{C_{tot}}, \tag{1}$$

where D_B and D_C – are individual diffusion coefficients of the mobile components - of free silicon and metal respectively, $C_{tot} = C_A + C_B + C_C + C_{Cp} + C_{Bt} + C_{Ct}$ – is the total concentration of all the components of the system.

Let us assume that the rate of destruction of silicon A at the points of the crystal lattice and the rate of generation of mobile silicon B are directly proportional to the concentration of silicon C_A at the points of its crystal lattice A and the concentration of metal C_C in silicon C. The equation describing the concentration of silicon C_A at the points of the crystal lattice A is presented as follows:

$$\frac{\partial C_A}{\partial t} = \frac{\partial}{\partial x}\left(D^* \frac{\partial C_A}{\partial x}\right) - k_1 \cdot C_C \cdot C_A. \tag{2}$$

Let’s assume that mobile silicon B participates in three processes:

– formation of silicon as a result of the destruction of the crystal lattice at a rate that is directly proportional to the concentration of atoms at the points of the silicon crystal lattice A and the concentration of atoms of the mobile metal C;

– diffusion of the metal film in silicon and the intergrain space by means of segregation trapping at a rate directly proportional to the concentration C_{Ct} of vacant traps Ct of the metal and the concentration C_B of mobile silicon B;

– trapping of the stationary complexes C_p in silicon at a rate directly proportional to the concentration of atoms of the mobile metal C.

Then, the equation for the concentration C_B of mobile silicon B can be presented as follows:

$$\frac{\partial C_B}{\partial t} = \frac{\partial}{\partial x}\left(D^* \frac{\partial C_B}{\partial x}\right) + \\ + k_1 \cdot C_A \cdot C_C - k_2 \cdot C_{Ct} \cdot C_B - k_3 \cdot C_B \cdot C_C. \quad (3)$$

Silicon is trapped in the intergrain space of the metal at a rate directly proportional to the concentration C_B of mobile silicon B and the concentration C_{Ct} of vacant traps $\mathrm{C_t}$. The equation for the concentration C_{Bt} of silicon Bt trapped in the intergrain space of the metal is

$$\frac{\partial C_{Bt}}{\partial t} = \frac{\partial}{\partial x}\left(D^* \frac{\partial C_{Bt}}{\partial x}\right) + k_2 \cdot C_{Ct} \cdot C_B\ . \quad (4)$$

The rate of trapping Ct in the intergrain space of the metal is directly proportional to the concentration of traps C_{Ct} and the concentration C_B of mobile silicon B. The equation for the concentration C_{Ct} of vacant trap centres $\mathrm{C_t}$ is

$$\frac{\partial C_{Ct}}{\partial t} = \frac{\partial}{\partial x}\left(D^* \frac{\partial C_{Ct}}{\partial x}\right) - k_2 \cdot C_B \cdot C_{Ct}\,. \quad (5)$$

The diffusion equation for the concentration C_C of the mobile metal C takes into account the fact that the metal is released from the traps in the intergrain space of the metal according to the exchange mechanism. It also takes into account the trapping of stationary complexes in silicon at a rate directly proportional to the concentration C_B of mobile silicon B:

$$\frac{\partial C_C}{\partial t} = \frac{\partial}{\partial x}\left(D^* \frac{\partial C_C}{\partial x}\right) + k_2 \cdot C_{Ct} \cdot C_B - k_3 \cdot C_B \cdot C_C\,. \quad (6)$$

The equation for the concentration C_{Cp} of metal atoms immobilised in $\mathrm{C_p}$ complexes takes into account the fact that the rate of bonding of mobile Me to the metal-silicon complexes is directly proportional to the concentration C_B of mobile silicon B and the concentration C_C of metal C in silicon:

$$\frac{\partial C_{Cp}}{\partial t} = \frac{\partial}{\partial x}\left(D^* \frac{\partial C_{Cp}}{\partial x}\right) + k_3 \cdot C_B \cdot C_C\,. \quad (7)$$

For equations (2)-(7), t – is the time, x – is the depth from the outer surface of the metal film, and k_1, k_2, and k_3 – are rate constants of the formation of mobile silicon, its trapping in the metal film, and formation of metal-silicon complexes respectively.

At the Me-Si interface, the condition for the reflection for all the components of the system is

$$\frac{\partial C_A}{\partial x} = \frac{\partial C_B}{\partial x} = \frac{\partial C_C}{\partial x} = \frac{\partial C_{Cp}}{\partial x} = \frac{\partial C_{Bt}}{\partial x} = \frac{\partial C_{Ct}}{\partial x} = 0$$

when $x = 0$ and $x = L$, (8)

where L is the thickness of the solution region in silicon.

The initial conditions for equations (2)–(7) when simulating the redistribution are the following:

$$C_\mathrm{A}(x, 0) = 0,\ \ C_{Ct}(x,0) = r{\times}N_\mathrm{SC},$$
$$C_\mathrm{C}(x,0) = (1-r){\times}N_\mathrm{SC},\ \ \text{when}\ \ 0 \le x \le h, \quad (9)$$

$$C_\mathrm{A}(x, 0) = N_\mathrm{SA},\ \ C_{Ct}(x,0) = 0,\ \ C_\mathrm{C}(x,0) = 0,$$
$$\text{when}\ \ h < x \le L, \quad (10)$$

$$C_\mathrm{B}(x, 0) = 0,\ C_{Bt}(x, 0) = 0,\ C_{Cp}(x, 0) = 0$$
$$\text{when all}\ \ 0 \le x \le L, \quad (11)$$

where h is the thickness of the metal film, N_SA is the intrinsic concentration of Si atoms ($N_\mathrm{SA} = 4.98{\cdot}10^{22}\ \mathrm{cm^{-3}}$), N_SC is the intrinsic concentration of metal atoms ($N_\mathrm{SC} = 5.68{\cdot}10^{22}\ \mathrm{cm^{-3}}$ for Ti, $6.34{\cdot}10^{22}\ \mathrm{cm^{-3}}$ for W, and $5.55{\cdot}10^{22}\ \mathrm{cm^{-3}}$ for Nb), and r is the number of traps for Si atoms in the intergrain space of Me.

The set of equations (2)–(7) with the concentration dependant interdiffusion coefficient (1) was solved numerically by means of the factorization method using implicit conservative difference schemes [17].

The model had the following parameters: individual diffusion coefficients of Me and Si, rate constants k_1, k_2, and k_3, and r.

Fig. 1 *a-c* shows experimental (points *1, 2*) and calculated (curves *1′, 2′*) concentrations of distributions of Me and Si along the depth of the Me-Si film system obtained by means of Rutherford backscattering spectroscopy (RBS) after magnetron deposition of Me (Ti, W, Nb) on single-crystal silicon and vacuum annealing at $T = 673$ K, $t = 30$ min. The experimental and calculated distributions were in good agreement when $k_1 = 1.{\cdot}10^{-26}\ \mathrm{cm^3/c}$, $k_2 = 1.{\cdot}10^{-18}\ \mathrm{cm^3/c}$, and $k_3 = 1.{\cdot}10^{-19}\ \mathrm{cm^3/c}$ were identical for all the metals used, and the model parameters were the ones given in Table 1. The obtained curves demonstrate that the dominating diffusant for all the studied systems is mobile silicon (curves *3′* in

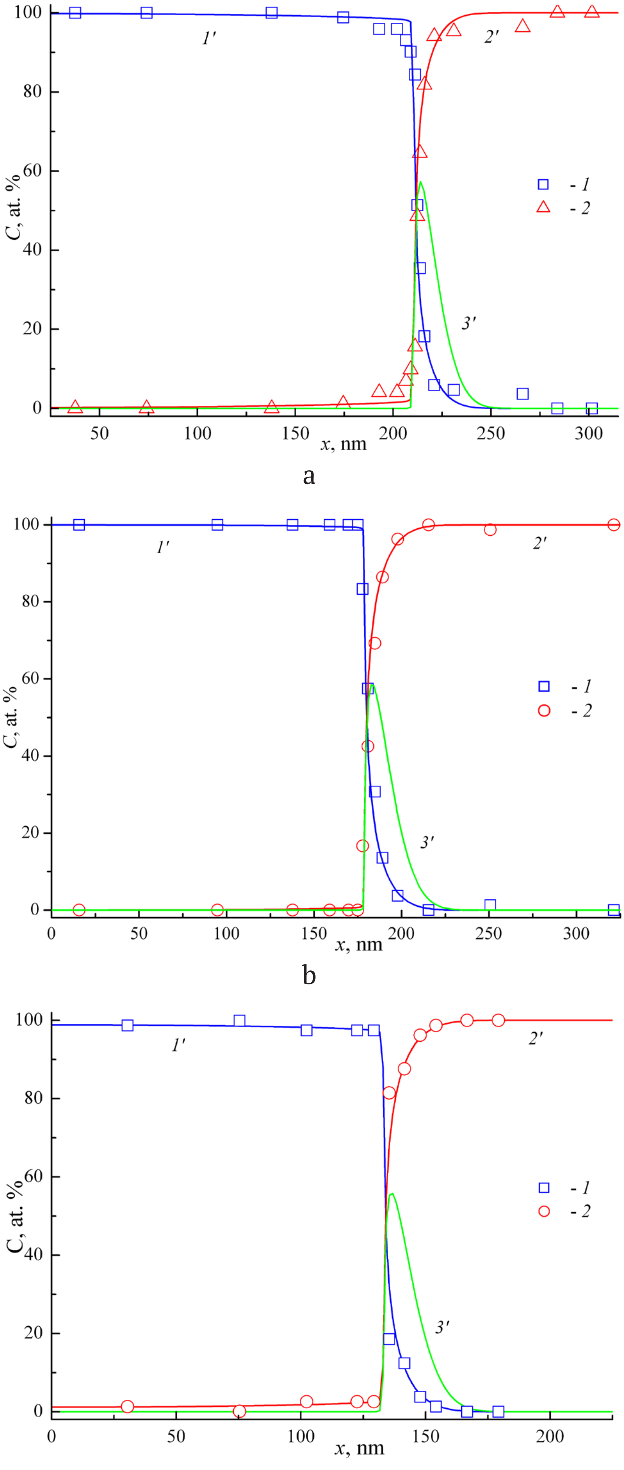

Fig. 1. Experimental (RBS) (points (symbols) *1, 2*) and calculated (curves *1′, 2′, 3′*) distributions along the depth of the Me–Si system of the total concentration *C* of metal (*1′*), silicon (*2′*), and its mobile atoms (*3′*). *1* – Me, *2* – Si; curves: *1′* – Me, *2′* – Si after magnetron sputtering and vacuum annealing at $T = 673$ K, $t = 30$ min. (*a*) – Ti, (*b*) – W, (*c*) – Nb.

Table 1. Parameters of the model for Ti, W, and Nb in the Me-Si system

Metal	$D_B \times 10^{14}$ cm^2/s	$D_C \times 10^{16}$ cm^2/s	r
Ti	4.0	4.0	0.02
W	1.0	6.0×	0.008
Nb	6.0	4.0	0.028

Fig. 1 *a-c*). Its distribution maximum is located at the Me/Si interface.

3. Conclusions

In our study, we developed a model of interdiffusion occurring during the formation of the polycrystalline metal film – single-crystal silicon system with limited solubility of the components. The model is based on the knowledge of solubility of silicon in the intergrain space of the metal, its segregation on the intergrain traps, and the solubility of metal in silicon limited by the complex formation process. Using the numerical analysis of the experimental distribution of concentrations of the elements of Me(Ti, W, Nb)–Si systems within the model, we determined the individual diffusion coefficients for metal and silicon, as well as the number of traps for Si atoms in the intergrain space of Me.

The model can be used to describe the redistribution of the elements of the Me-Si systems for the synthesis conditions ensuring the chemical interaction between metal and silicon and the formation of silicides. It also describes the mechanism of the possible formation of silicide phases near the Me/Si interface as the deep interdiffusion of the elements rather than layer-by-layer growth on the Me/Si interface.

The article also demonstrated that mathematical formalism suggested in [6] for the description of the interdiffusion in binary systems with unlimited solubility, can be used to describe the redistribution of the elements of Me-Si systems with limited solubility of the components.

The model can be used for empirical studies of the interdiffusion processes in Me-Si systems with limited solubility as well as to forecast the technological conditions for obtaining ohmic and non-ohmic Me-Si contacts in micro- and nanoelectronic devices.

Author contributions

Afonin N. N. – scientific leadership, research concept, simulation, text writing, final conclusions. Logachova V. A. – conducting research, review and text writing, editing the text.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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