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Interface interaction between thin films of transition metal compounds and silicon substrates across the native SiO_2 layer

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ABSTRACT

FeSi films deposited on the native oxide layer on silicon substrates have been shown to undergo a metal-like to insulator-like transition around the temperature of 250 K. Near room temperature the current was carried through the silicon substrate as a result of carriers tunneling across the film–substrate interface. This was facilitated by the formation of an ohmic contact to the silicon substrate across the SiO_2 layer. Rapid increase in the film–substrate interface resistance near 250 K switched the current to the film giving rise to a more than three orders of magnitude change in resistance. CoSi and Fe_2O_3 films deposited on silicon substrates also showed a similar transition in resistance near 250 K while resistance increased monotonically without any sign of a transition for TiSi and TiO_2 films. However, TiO_2 films doped with 6% Co behaved similar to films of Fe and Co compounds. These results conclude that the transition metal ions with multiple valences are important for the formation of an ohmic contact and the subsequent resistive transition. The magnitude of the transition was seen to depend on the energy of the depositing species during film growth. Ablation at high laser fluencies produced highly excited and ionized species. High density of interface states produced by energetic transition metal ions increased the hopping probability of carriers leading to a lower interface resistance. Formation of an ohmic contact between a metal electrode and the silicon substrate across the native SiO_2 layer in Metal/FeSi/SiO2/Si structures has been demonstrated.

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1. Introduction

Diffusion characteristics of metals in SiO_2 and Si have been of great interest as presence of metals lead to deterioration of device function and cause failure. Extensive research has been conducted on diffusion of metals such as Cu and Al in silicon that are readily used in metallization and metal–insulator–semiconductor (MIS) devices. Several reports also have been published on the diffusion characteristics of rare-earth metals and 3d transition metals [1,2]. These metals have been observed to cause both physical and chemical changes in SiO_2 and at the SiO_2/Si interface. Formation of impurity bands within the insulator, changes in band off-set at the SiO_2/Si interface, and reduction in SiO_2/Si surface work function were some of the physical changes that have been reported [3,4]. In addition, formation of silicides even at room temperature has been observed for transition metal elements with high diffusion [5].

We have reported the observation of an anomalous metal-like to insulator-like transition in FeSi films that were deposited on the native SiO_2 layer of a silicon substrate [6,7]. The resistivity of the p-type silicon substrate was seen to affect the magnitude of the resistance change near the transition point. FeSi films deposited on silicon substrates with both very high resistivity ($\sim 10^2\,\Omega$ cm at $10^{14}\,cm^{-3}$ doping) and very low resistivity ($\sim 0.01\,\Omega$ cm at $10^{19}\,cm^{-3}$ doping) did not show this transition while films on substrates with resistivity in the range of 1–10 Ω cm ($\sim 10^{17}\,cm^{-3}$ doping) showed the maximum change in resistance near the transition point. Near room temperature the resistance of the films decreased with decreasing temperature which resembled the metallic conductivity of the doped silicon substrate. Around a temperature of 250 K, the resistance increased by more than three orders of magnitude within a temperature range of about 20 K, followed by a gradual increase in resistance down to 50 K. The following mechanism was proposed to explain the observed metal-like to insulator-like transition in FeSi films. Near room temperature the carrier transport was seen to take place through the conducting silicon substrate which was enabled by impurity assisted tunneling across the interface. The diffusion of multi-valent Fe ions formed interface states that facilitated impurity-assisted hopping of carriers. For temperatures below about 250 K, the carriers became localized, perhaps due to the interaction between the tunneling electrons and the magnetic moment of the Fe ions at the interface, causing the resistance to increase. Below the transition temperature the carrier transport took place across

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the film, and at low temperature the mechanism of transport resembled that of variable range hopping. Along with the large change in resistance, more than 40% increase in magnetoresistance was also observed near the transition temperature [7]. This observation was attributed to the shrinking of electron wave functions by the applied magnetic field that decreased the probability of electron hopping between impurity ions [7,8]. In addition to FeSi films, we have investigated several other films of transition metal compounds, including CoSi, TiSi, Fe_2O_3, TiO_2, and 6% Co doped TiO_2, deposited on similar silicon substrates by the laser ablation technique. In this paper we present the transport properties of these films and the effect of the growth parameters on the metal-to-insulator transition. Furthermore, the tunnel current between a metal film and a silicon substrate across a native SiO_2 layer is expected to be very low. Inclusion of a thin FeSi layer between the metal film and the substrate alters the Fermi level shift at the interface and thus is expected to change the tunneling characteristics. Transport properties of Metal/FeSi/SiO_2/Si structures are also presented.

2. Experiment

Four different experiments were carried out to investigate the role of transition metal atoms at the SiO_2/Si interface on carrier transport. Films of silicides and oxides that contain transition metal atoms were grown by pulsed laser deposition (PLD) on boron-doped p-type silicon substrates with a resistivity in the range of 1–10 Ω cm. An excimer laser with a wavelength of 248 nm and a pulse width of 30 ns was used for ablation. Standard solvent cleaning techniques were used to prepare substrates for deposition. The native SiO_2 layer on silicon, which is estimated to be about 15–20 Å in thickness, was not removed prior to film growth. The temperature dependent transport properties of all the films were investigated by a four-point-probe technique in a closed cycle refrigeration system where temperature was varied from room temperature to 25 K.

Silicide film growth: Thin films of the transition metal silicides FeSi, CoSi, and TiSi, reported in this study were deposited by ablating compact powder targets of FeSi, CoSi, and TiSi, respectively. The deposition was carried out at a substrate temperature of 400 °C and a background pressure of about 10^{-6} Torr. Some films were also deposited at room temperature for comparison with films deposited at high temperature. Laser energy density at the target was 1.5 J/cm^2. The thicknesses of the films were in the range of 600–1000 Å.

Oxide film growth: The films of Fe_2O_3 and TiO_2 were deposited from compact powder targets of Fe_2O_3 and TiO_2. Cobalt doped TiO_2 targets were prepared by mixing TiO_2 and CoO_2 powders and sintering in high temperature to obtain 6% Co. Laser parameters used for oxide film growth were similar to those used in silicide film growth. All the oxide films were deposited at a substrate temperature of 400 °C and a background oxygen pressure of 10 mTorr.

Effect of laser fluence: Previously reported results on FeSi films deposited on silicon substrates [6,7] identified carrier transport across the film–substrate interface into silicon as the mechanism of transport near room temperature. To understand the role of Fe atoms/ions at the interface in the metal-like to insulator-like transition, we have controlled the depth of Fe atom diffusion at the interface during film growth by varying the energy of the Fe atoms impinging on the substrate. The energy of the Fe atoms arriving at the substrate was varied by changing the laser fluence at the target from 1.0 to 5 J/cm^2. An ion probe biased at a low negative voltage was used to obtain time-of-flight ion signals to calculate ion velocities and densities [9] at different laser fluences. Temperature dependent film resistance and IV characteristics of the films were determined by the four-point probe technique.

Metal films on silicon: This experiment was aimed at studying the interaction between a metallic film and the silicon substrate across an intermediate FeSi film. In the first step of the process FeSi films were deposited on a silicon substrate (native oxide layer intact) with a shadow-mask to form two isolated film regions. In the second step the substrate was moved to a sputtering chamber and aluminum or platinum films were deposited only on top of the FeSi film area (inset of Fig. 6) to form Metal/FeSi/SiO_2/Si junctions. Similar structures with Metal/SiO_2/Si junctions were also fabricated for comparison. A current and a voltage probe were placed on each of the metal pads. This film configuration eliminates the current path along the film and forces the current to propagate into the substrate across the interface between the film and the silicon substrate. This configuration enabled the probing of just the interface. The temperature dependence of the interface resistance and the IV characteristics were measured.

3. Results and discussion

3.1. Silicide films

The temperature dependence of resistance for FeSi, CoSi, and TiSi films deposited at a substrate temperature of 400 °C and that of a FeSi film deposited at room temperature are shown in Fig. 1. Both FeSi and CoSi films showed metallic behavior from room temperature down to about 260 K followed by a transition to an insulating state below this temperature while resistance of the TiSi film did not show noticeable temperature dependence. On the other hand, the FeSi films deposited at room temperature underwent a change in resistance of 10^5 Ω, which is an order of magnitude higher than the change in resistance observed for the high temperature deposited film. X-ray diffraction studies showed the films deposited at room temperature to be amorphous.

The resistivity of the p-type silicon substrates used in this study is 1–10 Ω cm, which corresponds to a boron doping concentration of about 10^{18}–10^{17} cm^{-3}. According to previous investigations the temperature dependent resistance of etched silicon substrates of this resistivity showed metallic behavior down to a temperature of 50 K [6]. That is consistent with the

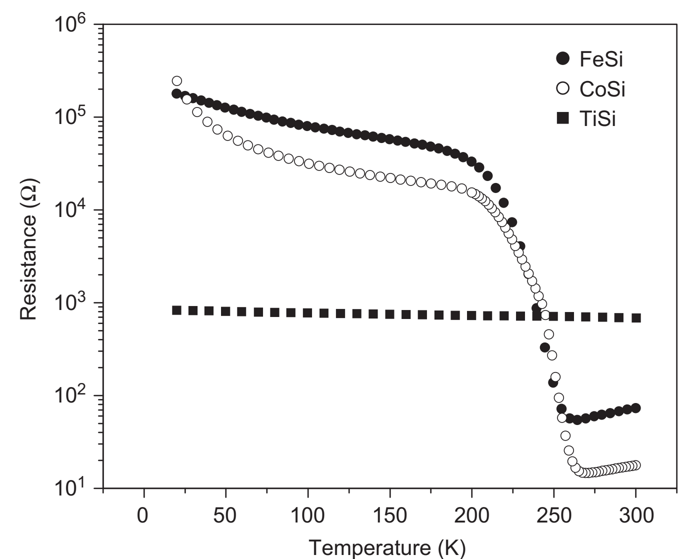

Fig. 1. Temperature dependence of the resistance of FeSi, CoSi, and TiSi films deposited at 400 °C on p-type silicon substrates with resistivity of 1–10 Ω cm.

results reported in the literature for highly doped silicon [10]. The metallic behavior resembling the conductivity of the silicon substrate observed for FeSi and CoSi films near room temperature can be explained by a mechanism of carrier tunneling into the substrate. In the presence of the native oxide layer, the pinning of the Fermi level at the interface leads to the formation of a depletion layer, which prevents carrier injection into the substrate. The width of the depletion layer (w) decreases with increasing substrate doping concentration (N), as given by the relation $w \sim 1/N^{1/2}$. As the transition metal ions diffuse across the interface, interface states and impurity levels (trap levels) are formed at the interface and within the depletion layer. For silicon substrates with relatively high doping concentrations where the depletion layer is narrow, transition metal impurity assisted tunneling of carriers across the SiO_2 layer and the narrow depletion layer causes the interface to be of low resistance. A good example of carrier transport via electron hopping between multivalent ions is the conductivity of transition metal doped glass [11,12]. A similar mechanism may account for the tunneling current between the film and the substrate in $FeSi/SiO_2/Si$ structures when the doping concentration of the Si substrate is relatively high. On the other hand, previous investigations have shown that the carrier tunneling into the substrate is absent for substrates with low doping concentrations, and the measured resistance resembles that of a FeSi film deposited on an insulating substrate [7]. This observation is also in agreement with the proposed mechanism. When the doping concentration of the substrate is low ($\sim 10^{15}\,cm^{-3}$) the depletion layer width becomes large and the probability of tunneling decreases, causing interface resistance to be high. The high resistivity of the interface confines the current transport to the film.

The observed monotonous increase in resistance with decreasing temperature in TiSi films (Fig. 1) suggests that the current transport is confined to the film at any temperature. For these films the diffusion of Ti has not facilitated carrier tunneling across the SiO_2 and the depletion layers. The main difference between the transition metals Fe, Co, and Ti is that Fe and Co ions are multivalent and possess magnetic moments while Ti exists mostly in Ti^{4+} state with a zero magnetic moment. This result supports the mechanism of carrier tunneling across the interface that is assisted by electron hopping between multivalent ions in FeSi and CoSi films. Hopping conduction is expected to be thermally activated and dependent on the distance between adjacent hopping sites [13,14]. The conductivity in V_2O_5 doped glass is an example of phonon-assisted electron hopping between the multivalent transition metal ions V^{4+} and V^{5+}. The conductivity of these materials is given by [11],

$$\sigma = \left(\frac{v_0 N e^2 R^2}{kT}\right) c(1-c)\exp(-2\alpha R)\exp\left(\frac{-W}{kT}\right) \qquad (1)$$

where v_0 is the optical phonon frequency, N is the density of the transition metal ions, e is the electric charge, R is the average separation between two transition metal ions, c is the ratio of the number of ions in the low and high valence states, α is the rate of decay in the radial wave function, and W is the activation energy.

With decreasing temperature the probability of electron localization at the impurity atoms increases exponentially. Rapid increase in interface resistance near 250 K may result from such an electron localization mechanism.

Transition metal oxide films: Temperature dependent resistance of Fe_2O_3, TiO_2, and 6% Co doped TiO_2 films deposited on silicon substrates are shown in Fig. 2. The mechanism of carrier transport in Fe_2O_3 films is similar to that in FeSi films. The preferred path of current transport near room temperature is through the silicon substrate, which is facilitated by multivalent Fe ion assisted

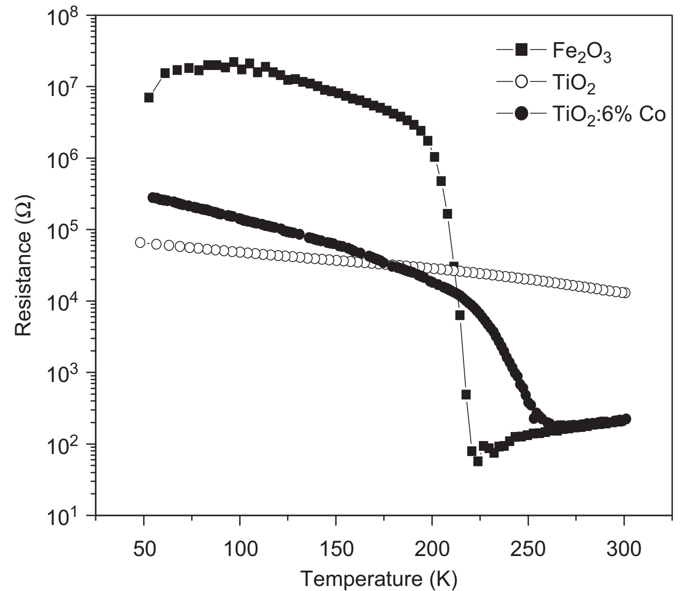

Fig. 2. Temperature dependence of the resistance of Fe_2O_3, TiO_2, and 6% Co doped TiO_2 films deposited at 400 °C on p-type silicon substrates with resistivity of 1–10 Ω cm.

tunneling across the interface. Similar to FeSi films, localization of electrons at Fe sites increased interface resistance around a temperature of 225 K, forcing the current to flow in the highly resistive Fe_2O_3 film. The reason for the lower transition temperature in Fe_2O_3 films in comparison to FeSi films is not clear. It has been reported that in absence of an oxide layer diffusion species at a Fe/Si interface is Si while in presence of an oxide layer Fe becomes the diffusion species, leading to the formation of Fe–Si bonds at the interface [15]. In presence of a higher concentration of oxygen, formation of Fe–O bonds at the SiO_2/Si interface has been observed [16]. The tunneling of the charge carriers across the interface that is assisted by the Fe impurities is expected to be affected by the changes in the nearest neighbor interaction of the Fe atoms.

Similar to TiSi films, carrier tunneling into the silicon substrate at high temperature was not observed in TiO_2 films. However, the presence of a small percentage of Co in the film was sufficient to evoke the carrier tunneling mechanism near room temperature. Similar to FeSi and CoSi films, carrier transport switched over to the film below a temperature of 250 K. This result clearly demonstrates the role of multivalent transition metal atoms in the conduction mechanism of these films.

Effect of laser fluence: Our previous results suggested that the interface states produced by the diffusion of Fe and Co ions to the interface are responsible for the transport mechanism across the interface at room temperature. In this experiment we have affected the diffusion characteristics by varying the laser fluence of the ablation laser. Fig. 3 shows the results from the ion–probe study that describes the variation of the ion density and the ion energy with increasing laser fluence. The kinetic energy of the ions was computed based on the most probable velocities derived from the time-of-flight ion probe signals while the density of ions was obtained by computing the area under the time-of-flight ion profiles. The ion density begins to saturate beyond a fluence of 3 J/cm^2 while kinetic energy of the Fe ions increased from 2.3 eV at 1 J/cm^2 to 76 eV at 5 J/cm^2. For a laser energy density that is above the ablation threshold of the target material, plasma of the evaporated material is formed during the laser–target interaction. With increasing laser fluence the early part of the laser pulse forms the plasma while the latter part of the pulse is absorbed

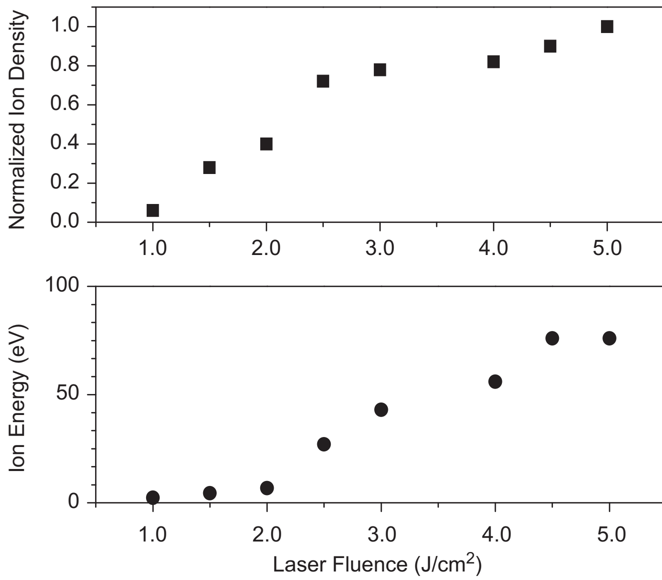

Fig. 3. Dependence of ion density and ion energy on the energy fluence of the ablation laser measured by an ion probe placed 6 cm from the target.

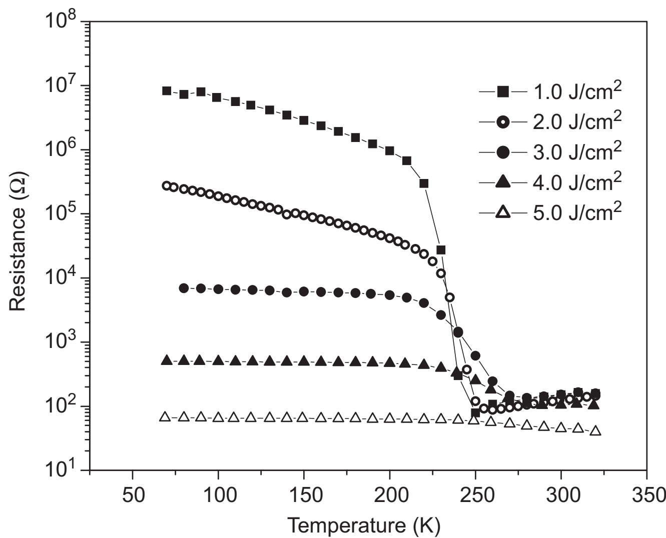

Fig. 4. Temperature dependence of the resistance of FeSi films deposited with laser fluencies in the range of 1–5 J/cm^2. Films were deposited at 400 °C on p-type silicon substrates with resistivity of 1–10 Ω cm.

into the plasma by the inverse-bremsstrahlung process [17–19]. Above a certain fluence the target is screened by the generated plasma and no new material is ablated from the target. However, absorption of radiation into the plasma increases the plasma temperature, which in turn increases the ion density. The inverse-bremsstrahlung absorption continues till the plasma frequency exceeds that of the laser radiation, at which point the plasma become reflective [17]. Subsequent to the termination of the laser pulse the plasma expands adiabatically transforming initial thermal energy into kinetic energy. The higher the laser fluence, the higher the density and the kinetic energy of the ions. Therefore, the atoms and the ions that impinge the substrate at high laser fluencies are expected to have higher diffusion lengths.

Temperature-dependent resistance of FeSi films deposited on silicon substrates with laser fluencies in the range of 1–5 J/cm^2 are shown in Fig. 4. The low resistance near room temperature due to carrier tunneling into the silicon substrates is common to all the films. The main difference was observed in the insulator-like state after the transition near 250 K. A simple model that considers two parallel paths of current transport can be used to describe the experimentally measured resistance. One path will be along the film with resistance R_1 while the other path contains the following three steps: across the interface into the substrate (resistance R_2), along the substrate (resistance R_3), and across the interface into the film (resistance R_2). The measured resistance R from the four-point probe date can be expressed as $R = R_1(2R_2+R_3)/(R_1+2R_2+R_3)$. The temperature dependence of R_1 and R_3 were obtained by four-point probe measurements of a FeSi film on an insulating substrate and of an etched silicon substrate, respectively. The temperature dependence of the interface resistance R_2 required to produce the measured resistances of the film for the five different laser fluencies were determined using this simple model. To account for the results presented in Fig. 4, R_2 in the temperature range of 250–220 K should change by factors of 10^4, 10^3, 20, and 4 for films deposited at the fluences of 1, 2, 3, 4, and 5 J/cm^2, respectively. The consistent reduction in the interface resistance with increasing energy and density of the deposited Fe atoms/ions supports the concept of Fe or Co ion

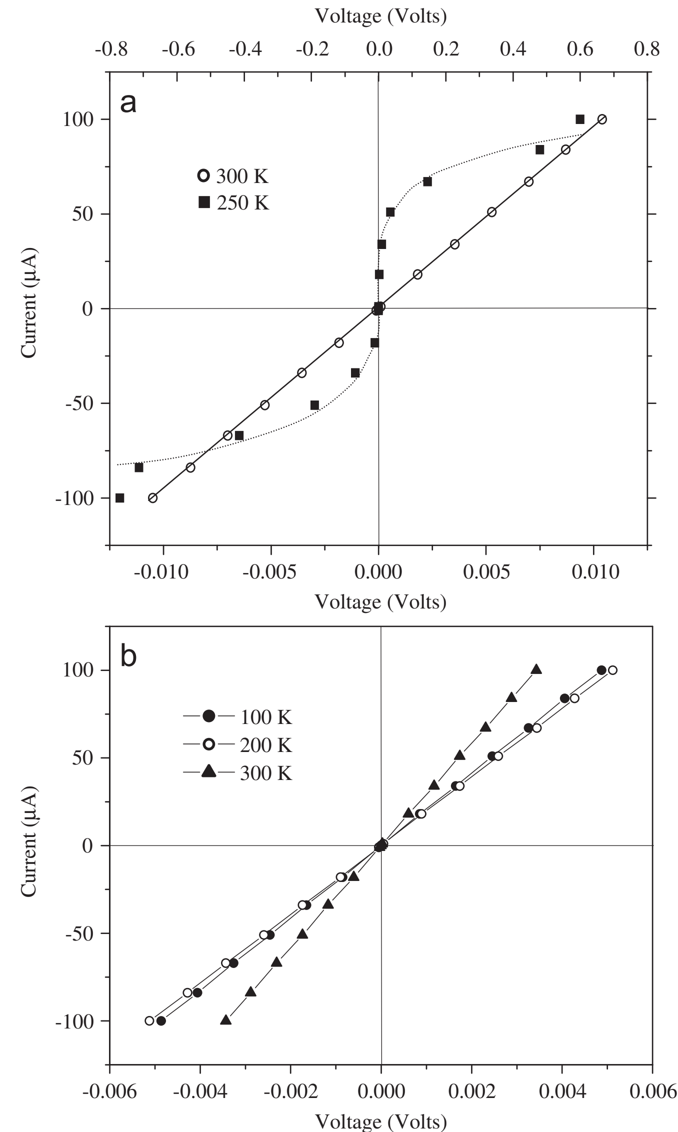

Fig. 5. *I–V* characteristics at different temperatures for FeSi films deposited with laser fluencies of (a) 1 and (b) 5 J/cm^2.

assisted tunneling as the mechanism for producing low resistance near room temperature. Increasing impurity density (N) decreases separation between impurities (smaller R) and thus, according to Eq. (1), the overall conductivity increases.

The IV characteristics of films deposited at 1 and 5 J/cm^2 fluences are compared in Fig. 5. Near room temperature all the films showed linear IV characteristics that are indicative of an ohmic contact between the film and the silicon substrate. This observation is consistent with a tunneling mechanism that is independent of temperature. However, the nonlinear IV characteristics observed after the metal-like to insulator-like transition in films deposited at low fluencies, (Fig. 5(a)), suggest a change in the conduction mechanism. The films deposited at high fluence maintained an ohmic contact with the silicon substrate at all temperatures (Fig. 5(b)).

Metal films on silicon: The metal and metal/FeSi films deposited on silicon substrates with the native SiO_2 layer were formed as two isolated pads. The only path of current transport in the four-point-probe measurement was across the SiO_2/Si interface. Therefore, the measured IV characteristics provide information on the mechanism of transport at the interface. The temperature dependence of the measured resistance for Al/SiO_2/Si, Al/FeSi/SiO_2/Si, Pt/SiO_2/Si, and Pt/FeSi/SiO_2/Si film structures are shown in Fig. 6. The rapid increase in resistance followed by a gradual increase in resistance below about 250 K observed in both Al/SiO_2/Si and Pt/SiO_2/Si structures indicate different mechanisms of transport. Plots of resistance vs. $1/T$ and $1/T^{1/4}$ in the temperature ranges of 300–130 K and 130–40 K, respectively, showed nearly linear behavior (Fig. 7). The two different linear regimes in Fig. 7(a) correspond to thermally activated hopping mechanisms via localized states that are commonly observed in noncrystalline semiconductors [14]. The first regime resembles the conductivity due to excitation of carriers from impurity states near the Fermi level to localized states near the band edge (E_c) of a wide band gap material, followed by hopping of carriers between localized states at the band edge. The conductivity resulting from this mechanism is given by

$$\sigma = \sigma_0 \exp\left\{-\frac{E_A - E_F + W_1}{kT}\right\}$$

where E_A is mobility edge that describes the energy of the localized states at the conduction band edge, E_F is the Fermi energy, and W_1 is the activation energy for hopping [14]. The second mechanism of conduction is the thermally activated hopping of electrons between two localized levels near the Fermi level. For an insulator such as SiO_2, Fermi energy will be near the middle of the band gap. The conductivity due to this mechanism is represented by near-neighbor hopping given by

$$\sigma = \sigma_0 \exp\left\{-\frac{W_2}{kT}\right\}$$

where W_2 is the hopping energy [14]. With decreasing temperature, near-neighbor hopping weakens and gives rise to variable-range hopping conduction that is described by

$$\sigma = \sigma_0 \exp\left\{-\frac{B}{T^{1/4}}\right\},$$

where B is a constant [14]. The results presented in Fig. 7(b) agree well with variable-range hopping conduction.

The presence of the thin FeSi layer in Al/FeSi/SiO_2/Si and Pt/FeSi/SiO_2/Si structures reduced the interface resistance by more

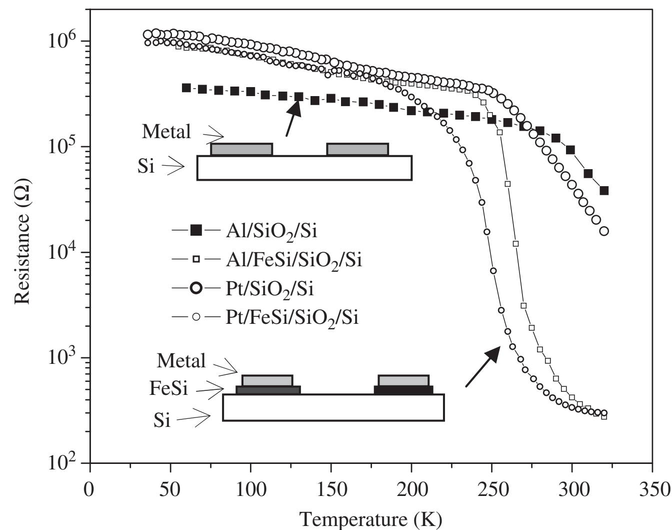

Fig. 6. Temperature dependence of the resistance of Al/SiO_2/Si, Al/FeSi/SiO_2/Si, Pt/SiO_2/Si, and Pt/FeSi/SiO_2/Si structures. Configuration of the discontinuous film layers is shown in the inset.

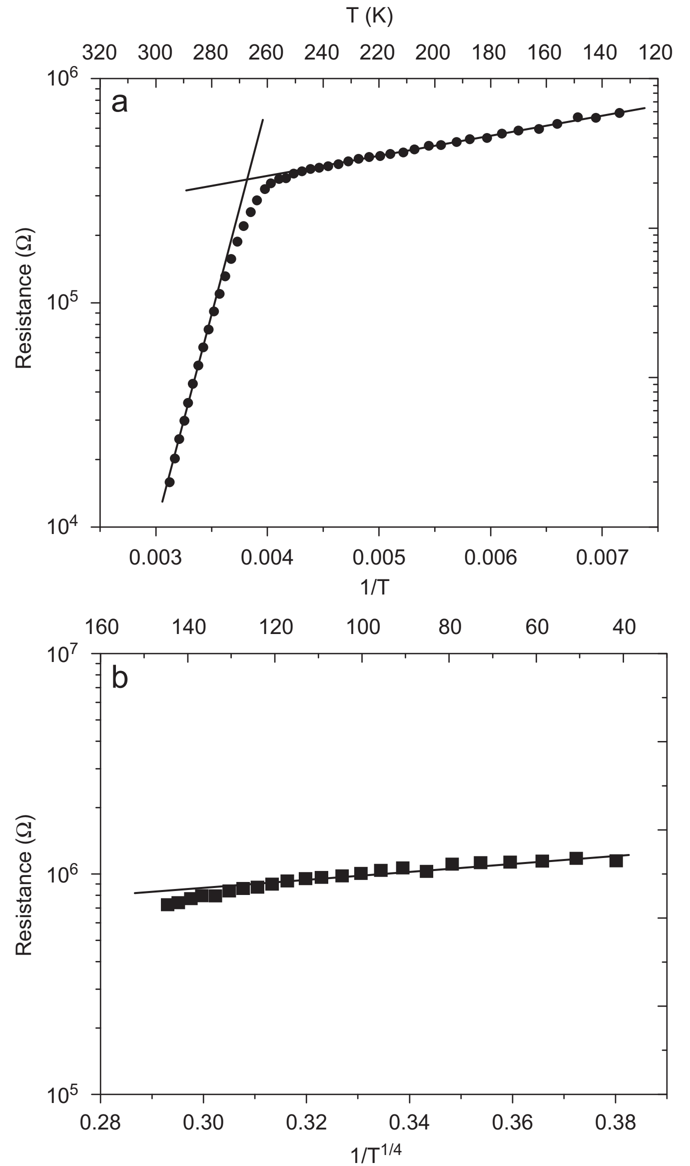

Fig. 7. Variation of resistance in Pt/SiO_2/Si structures as a function of (a) $1/T$ in the temperature range of 320–140 K, and (b) as a function of $1/T^{1/4}$ in the temperature range of 140–40 K.

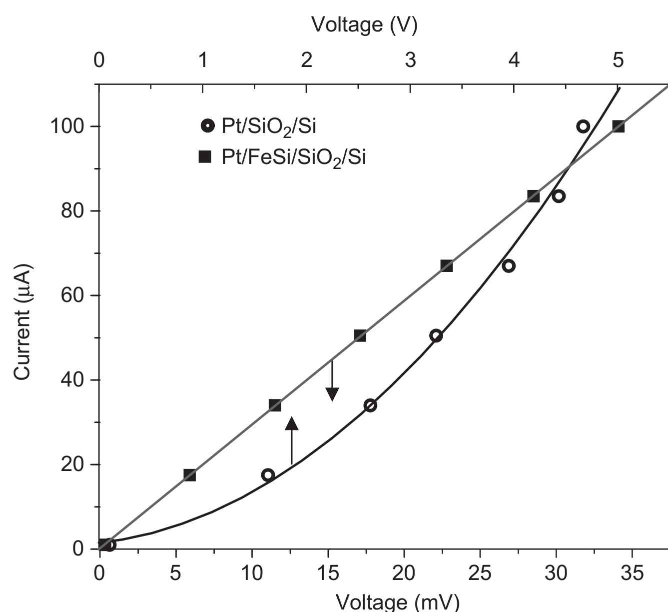

Fig. 8. IV characteristics of the structures Pt/SiO_2/Si and Pt/FeSi/SiO_2/Si at 300 K.

than two orders of magnitude. The IV characteristics presented in Fig. 8 shows the formation of an ohmic contact near room temperature between the Pt electrode and the silicon substrate across the SiO_2 layer when a thin (~50 nm) FeSi film is present between the substrate and the metal film.

In summary, a metal-like to insulator-like transition near a temperature of 250 K has been observed in FeSi, CoSi, and Fe_2O_3 films that were deposited on conducting silicon substrates with the native SiO_2 layer intact. The metallic behavior near room temperature results from carrier tunneling into the substrate via a mechanism of carrier hopping between the multivalent transition metal ions. The onset of charge carrier localization near 250 K increased the resistance at the film–substrate interface forcing the current to flow on the film. The absence of such a transition in TiSi and TiO_2 films and the appearance of the transition when TiO_2 is doped with 6% of Co illustrate the importance of the presence of multivalent transition metal ions at the interface to enable tunneling of charge carriers across the interface near room temperature. The diffusion of Fe ions to the film–substrate interface has been manipulated by the energy of the ablation laser. High ablation laser energy corresponds to high density of Fe ions at the interface. The reduced distance between impurity ions increases hopping probability leading to an ohmic contact between the film and the substrate at all temperatures. The current transport between a metal contact and the silicon substrate across the Metal/SiO_2/Si structures followed three distinct mechanisms. Two mechanisms were thermally activated with different activation energies while at low temperature conduction followed a mechanism of variable range hopping. When the metal contact was deposited on a FeSi film, the favored Fe ion assisted carrier hopping near room temperature enabled the formation of an ohmic contact between the metal electrode and the silicon substrate across the insulating native SiO_2 layer.

Acknowledgments

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