Composition analysis of nickel silicide formed from evaporated and sputtered nickel for microsystem devices

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Nickel silicide (NiSi) is highly suitable for microsystem fabrication, exhibiting suitable mechanical properties and good resistance to bulk micromachining etchants. Conditions for the formation of nickel silicide by vacuum annealing thin films of nickel deposited on silicon substrates are investigated. Nickel silicide thin films formed using sputtered and evaporated nickel films were analysed using Auger electron spectroscopy, which has shown that evaporated thin films of nickel tend to form nickel silicide more readily and with less thermal effort.

Introduction: Nickel silicide (NiSi) has good mechanical properties and can be used to form micro-structures such as cantilevers, bridges, and membranes [1]. Formation of micro-structures can involve bulk micromachining of silicon using etchants such as potassium hydroxide (KOH) and tetra-methyl-ammonium hydroxide (TMAH). NiSi has a low etch rate of 0.75 nm/min in 25% KOH and does not etch in 25% TMAH [1].

Nickel silicide growth involves the consumption of silicon, when it is formed by thermally reacting deposited nickel on silicon substrates. In this Letter we compare the composition of thin films formed as a result of reacting sputtered nickel and evaporated nickel on silicon. Auger electron spectroscopy depth profiles have been used to study the composition of the thin films formed.

Nickel silicide formation: Thin films (50 nm) of nickel on (100) n-type silicon substrates were subjected to different vacuum annealing conditions in order to form nickel silicide of the desired monosilicide composition (1:1 nickel and silicon). The nickel thin films were either deposited by DC magnetron sputtering or by electron-beam evaporation. Sputtering was carried out under the conditions listed in Table 1 and evaporation of 50 nm of nickel was performed from 99.99% pure nickel sources, after pumping down to a base pressure of 2×10^{-7} Torr.

Table 1: DC magnetron sputtering conditions

Target	Nickel (99.99%)
Target diameter	100 mm
DC power	80 W
Target to substrate distance	50 mm
Process gas	Argon 5.0
Base pressure	1.0×10^{-5} Torr
Sputtering pressure	1.0×10^{-2} Torr
Sputtering duration	60 s

Formation of nickel silicide (NiSi) from silicon takes place through a series of stoichiometric transformations [2]. Nickel thin films on silicon react to form Ni_2Si at about 250°C, NiSi at 350°C, and $NiSi_2$ above 650–700°C. Patterned nickel samples on (100) n-type silicon were subjected to a one-step or two-step contact anneal process. The following temperatures were among those used to anneal both evaporated and sputtered nickel films (50 nm) on silicon: (i) 200°C for 1 h, followed by 350°C for 3 h; (ii) 250°C for 1 h, followed by 350°C for 3 h; (iii) 250°C for 1 h, followed by 400°C for 3 h. The samples were placed on a substrate heater in a vacuum chamber. The anneal process was started under vacuum of 1.0×10^{-5} Torr. The samples were then allowed to cool in vacuum.

Results: The resulting films were analysed using Auger electron spectroscopy (AES) depth profiles carried out on a VG310F scanning Auger microprobe. A full spectrum (at a constant retard ratio of 4 and a beam voltage of 10 kV) was collected after each sputter etch cycle and the depth profiles against argon ion sputter time were constructed from the measured peak areas using the library sensitivity factors in the Avantage software package. Depth profiles show that the sputtered nickel films annealed using conditions (i), (ii), and (iii) did not react uniformly with silicon (Fig. 1). The AES depth profile of sputtered nickel films vacuum annealed at 600°C for 90 min (not shown) shows more complete reaction with silicon, giving NiSi with a composition of 48% nickel to 52% silicon.

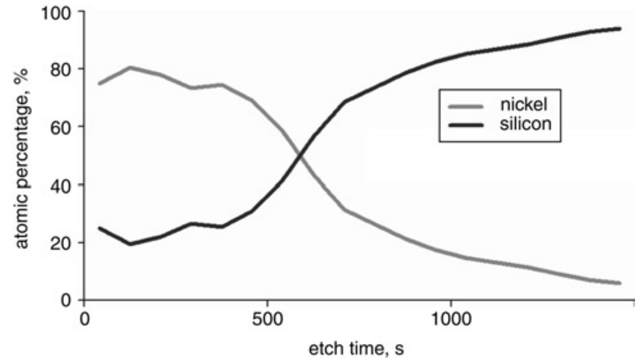

Fig. 1 *Auger electron spectroscopy depth profile for sputtered nickel on silicon vacuum annealed at 250°C for 1 h, followed by 350°C for 3 h*

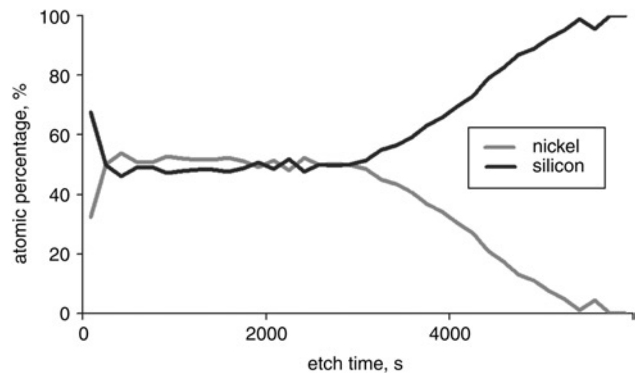

Fig. 2 *Auger electron spectroscopy depth profile for evaporated nickel on silicon vacuum annealed at 350°C for 30 min*

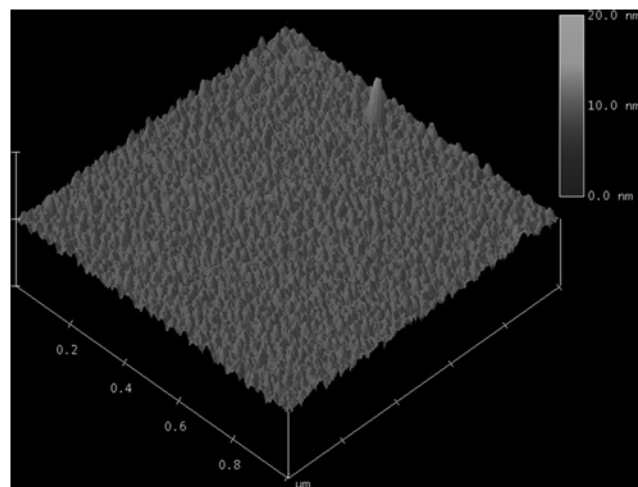

Fig. 3 *Atomic force microscope surface scans over 1 × 1 µm area of nickel silicide formed from evaporated nickel on silicon by vacuum annealing at 350°C for 30 min*

Evaporated nickel films reacted using the same conditions formed NiSi films, with the composition of the silicide being 49–50% nickel and 51–50% silicon. Evaporated nickel films were also vacuum annealed at 350°C for different time periods of 30 min and 1 h. Depth profiles of these films (formed using 50 nm evaporated nickel) showed them to be NiSi (with nickel and silicon ratios of almost 50:50). Fig. 2 depicts the depth profile obtained for a NiSi film which was formed after 30 min of vacuum annealing at 350°C. The depth profiles of NiSi films formed with evaporated nickel using conditions (i), (ii), and (iii) were similar to Fig. 2.

Atomic force microscope (Digital Instruments Dimension 3100) surface scans of NiSi thin films formed from evaporated nickel (350°C for 30 min) showed that their average surface roughness was

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found to be 0.67 nm (Fig. 3), compared to 9.77 nm for the silicide formed at 600°C from sputtered nickel.

Discussion: Nickel silicide films formed by vacuum annealing of electron-beam evaporated nickel were found to be NiSi, with the composition being very close to 50% nickel and 50% silicon. For evaporated nickel films, all reported temperature and time conditions resulted in films of required composition. No traces of oxygen were found in any of the films. The measured step-height of nickel silicide film above the surface of silicon, the total amount of nickel silicide formed, and the measured thin film resistivity values of 14 μΩ-cm correspond very well to the values reported in the published literature [3].

Compared to evaporated nickel films, it was found that more thermal effort was required to form NiSi using sputtered nickel films. Only sputtered nickel films which were annealed at a high temperature of 600°C appeared to have completely reacted. This could be attributed to variations in the diffusivity of metals in silicon, based on the metal thin film deposition technique [4].

Conclusion: Conditions for the formation of nickel silicide thin films, for use in microsystem fabrication, have been discussed. Nickel silicide films formed by vacuum annealing of sputtered and evaporated nickel films have been compared using Auger electron spectroscopy depth profiles. Nickel silicide of NiSi composition was formed using evaporated nickel using several annealing temperatures and time periods. Higher thermal energy was required to form NiSi using sputtered nickel films, which did not react uniformly with silicon at lower temperature conditions.

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