Optimisation of a smooth multilayer Nickel Silicide surface for ALN growth

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Abstract. For use in thin film electroacoustic (TEA) technology a few hundred nanometre thick nickel silicide (NiSi) electrode would need to be fabricated. A complete fabrication process for the formation of over 200 nm thick silicide films has been optimised for use as an electroacoustic electrode. Optimisation of silicidation temperature and identification of the mono phase of silicide is demonstrated. Thick electrodes are formed by depositing multilayers of silicon and nickel pairs onto silicon (Si) substrates before rapid thermal annealing. The numbers of multilayers and relative material thicknesses are optimized for both surface roughness and electrical resistivity. The growth of textured aluminium nitride (AlN) has been investigated on the optimised surfaces.

1. Introduction

The use of metal silicides in integrated circuits as a contact material has been widely researched [1]. Nickel silicide (NiSi) combines the advantages of low electrical resistivity and a low formation temperature for the mono-silicide phase which is stable between 300°C and 700°C. This makes NiSi an attractive candidate for electrode replacement in view of promoting IC compatibility with TEA technology. As an electrode material NiSi enables access to front end integration between IC and electroacoustic (EA) technologies with advantages for high temperature sensors. Further benefits would include reduced losses by elimination of the soft metals currently used as electrode materials.

In this work a new fabrication method is optimised for the production of thick NiSi electrodes with an arbitrary thickness. The process developed to bring about an arbitrary thickness involves the manufacture of a multilayer stack of alternating Si and Ni layers followed by a thermal treatment. The layers in the stack have been optimised for deposition thickness, which has brought improvements in silicide resistivity and a decrease in surface roughness. Growth of textured AlN on the prepared silicides is investigated with a corresponding improvement observed for smoother surfaces, 7.5° to 3.5° full width half maximum (FWHM) of the rocking curve respectively.

2. Experimental

Initial silicide films were optimised for monosilicide formation by sputter deposition of 20 nm nickel films onto p-Si (100) substrates. Subsequent rapid thermal annealing (RTA) was performed at temperatures between 200°C and 550°C. Four point probe measurements of the sheet resistance were measured before RTA, after RTA and after etching of the silicide films in 100% HNO₃, the latter to remove any excess nickel. Phase determination of the resultant silicides was performed by analysis of
the sheet resistance and X-ray diffractograms (XRD) which were taken using a Phillips X’pert MRD diffractometer.

After temperature optimisation, four multilayer stacks were investigated containing 3, 5, 6 and 10 multilayer pairs, each multilayer stack having a combined deposition thickness of 300 nm. The multilayer pairs were formed by sputter deposition of silicon followed by sputter deposition of nickel. The whole stack of multilayer pairs was formed without breaking vacuum. The ratio in each layer of silicon deposited to nickel was 2:1. Prior to deposition the system was gettered by means of a ten minute Ti pre-sputter. All multilayer pairs investigated were sputter deposited onto cleaned n-type Si (111) wafers. Sheet resistance was again measured prior to RTA at 500°C, after annealing and after removal of excess Ni. A 2 μm thick AlN film was deposited by reactive sputtering using a Von Ardenne reactive balanced magnetron sputter deposition system operated in a pulsed direct-current (DC) mode. The target was powered by a ENI RPG50 asymmetric bipolar-pulsed DC generator. The applied frequency was 250 kHz, while the duration of the positive pulse was 500 ns. The system is evacuated by a 800 1/s turbo molecular pump achieving a base pressure of <5.10^{-8} Torr prior to deposition. The AlN films were sputter deposited at room temperature [2] with deposition parameters given elsewhere [3]. The influence of the NiSi film on the textured growth of AlN was investigated with XRD.

3. Optimisation

Optimisation of the nickel silicide RTA temperature was performed to choose the low resistivity monosilicide phase and RTA time was held constant at 30s. No noticeable difference between sheet resistances prior to and after RTA was seen, which indicates that all the deposited nickel had been consumed. The results are shown in figure 1(a), were the transition between the high resistivity nickel rich Ni₃Si phase and the low resistivity NiSi can be seen to occur at temperatures between 250°C and 300°C. However, complete phase transition of the silicidation does not seem to occur until 400°C, where the sheet resistance remains fairly consistent.

![Figure 1](image)

**Figure 1.** (a) Sheet resistance of thin NiSi films as a function of RTA temperature. (b) Diffractograms from three nickel silicide films for RTA temperatures showing the transition between the first two silicide phases, Ni₃Si and NiSi, as well as at the optimised temperature.

This can also be seen in figure 1(b) where the diffractogram for 250°C RTA shows the (220) (121) and (002) Ni₃Si planes. Only the (211) monosilicide plane is visible at this silicidation temperature, whilst at 300°C the (013) NiSi plane is also seen. An optimum sheet resistance of 2.3 Ω/sq is obtained at 500°C with three monosilicide planes, including the (112) plane, visible in the XRD diffractogram.

Formation of thick nickel silicide films is achieved via sputter depositing silicon and nickel pairs in the multilayer stack and then RTA annealed at 500°C for 30s to achieve complete transformation to
the low resistive NiSi phase. The thickness required can be calculated by using the published ratio of resultant NiSi thickness to total deposited nickel thickness [4]. In this case 100 nm total of Ni is deposited giving a film thickness around 200 nm for mono-silicide. The deposited thicknesses for each pair and the number of multilayers needed to achieve this silicide thickness are shown in Table 1.

Atomic force microscopy (AFM) was used to determine the RMS value of the surface roughness of the resultant silicides after RTA and removal of any excess nickel. No noticeable difference between sheet resistance before and after RTA indicated the consumption of all deposited nickel. The results of the measured surface roughness are given in figure 2 indicating very smooth surfaces for an increased number of multilayers. Increasing the number of layers in multilayer stack requires thinner silicon and nickel layers to obtain the same overall thickness. For thinner layers the main diffusing species, nickel, has a decrease in diffusing distance needed to form NiSi than for multilayer stacks with thicker layers. For the three-layer stack the distance for the diffusing species to travel to form NiSi includes a multilayer pair of around 100 nm which could in turn leave increased undulation of the film surface. Investigations of this phenomenon were performed by depositing the same multilayer stack but with the nickel and silicon thicknesses reversed. Once more it was observed that the thicker deposition pairs resulted in increased RMS values of the surface roughness, over 6 nm for the 3 layer stack. For all multilayer stacks here the surface roughness increased, probably due to Ni diffusion to and consumption of Si in the substrate. In all cases the surface roughness was around 0.3 nm prior to RTA.

| No. Layers | Si (nm) | Ni (nm) | Total (nm) |
|------------|---------|---------|------------|
| 3          | 66      | 33      | 297        |
| 5          | 40      | 20      | 300        |
| 6          | 32      | 16      | 288        |
| 10         | 20      | 10      | 300        |

Table 1. Number of layers, their respective deposition thicknesses and total thickness deposited.

There is a reduction in surface roughness to around 1 nm RMS for the ten-layer system, which compares favourably to that reported elsewhere for 30 nm of nickel deposited directly onto Si substrates [5]. As seen in figure 2, the sheet resistance remains fairly consistent for each multilayer stack and scales favourably with sheet resistances shown earlier for 20 nm nickel depositions on plain Si wafers. Again the 10 layer system indicates an optimum sheet resistance close to 0.4 Ω/sq.

Investigations of the growth of AlN on NiSi were performed for the 5 and 10 multilayer systems. In figure 3(a) it can be seen that there is an influence on AlN growth from the NiSi surface. For both silicide surfaces textured growth of the (002) plane of AlN is achieved. However, AlN deposition on the 10 multilayer stack demonstrates increased textured growth of the (002) plane. This is confirmed with rocking curves measurements, figure 3(b), which show that the texture of the AlN film grown onto 10-layer NiSi shows a significant improvement, from 7.5° to 3.5°, in the full width half maximum (FWHM) of the (002) rocking curve. Influence on the growth mechanism of AlN has been investigated previously with influences on AlN growth from crystallographic influence of the underlying layer and surface roughness [6]. The NiSi wafer samples were diced so that all AlN
depositions were performed at the same time and with a Si reference sample which indicated a high quality 2° FWHM (002) AlN deposition.

![Diffractograms of textured AlN deposited on thick monosilicide films utilising the 5 and 10 multilayer stack. (b) FWHM of the (002) rocking curve, for AlN deposited on a 5 and 10 multilayer stack after formation of NiSi.](image)

Figure 3. (a) Diffractograms of textured AlN deposited on thick monosilicide films utilising the 5 and 10 multilayer stack. (b) FWHM of the (002) rocking curve, for AlN deposited on a 5 and 10 multilayer stack after formation of NiSi.

Investigation of AlN grown onto the NiSi, with the Si and Ni thicknesses reversed and a surface roughness of 6 nm, demonstrated a saturation of the FWHM of (002) AlN at 7°. Thus the dominating influence on AlN growth should be due to crystallographic influence of the silicides. It can be seen in figure 4 that the 10-layer system includes the (112) and (013) NiSi planes which may positively influence the AlN growth. This however is yet to be satisfactorily confirmed with AlN grown on other silicides.

4. Conclusions
An optimization process for the fabrication of thick NiSi has been demonstrated. Formation of low resistive monosilicide with a very smooth surface has been achieved. A method for producing thick NiSi films with an arbitrary thickness has been presented and textured growth of c-axis orientated AlN on silicides has been achieved, showing 3.5° FWHM. Further research is being conducted using Ti seed layers and using TEM studies to understand the growth mechanism of AlN on NiSi. New FBAR resonators are in production which will be analyzed with extraction of NiSi acoustic properties.

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