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Three-dimensional composition mapping of NiSi phase distribution and Pt diffusion via grain boundaries in Ni_2Si

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The formation of Ni silicide alloyed with Pt has been analyzed by atom probe tomography. A 300 °C/1 h anneal results in simultaneous growth of the NiSi and Ni_2Si phases: the Ni_2Si phase is a continuous layer with columnar grains, while the NiSi phase forms a discontinuous layer. Direct evidence of Pt diffusion short-circuits via Ni_2Si grain boundaries is shown. The presence of Pt in the grains and interphase boundaries may explain the change in the Ni silicide formation for the Ni(5% Pt)/Si system.

Keywords: Ni silicide; Diffusion; Grains boundary; Atom probe; Platinum

Metal silicides are widely used as contacts and interconnections to source and drain structures in microelectronics devices. NiSi is the best silicide for contact material in advanced integrated circuits but its high-temperature stability is of concern [1]. It has been shown that the addition of Pt in the Ni film increases the nucleation temperature of $NiSi_2$ by approximately 150 °C and thus stabilizes the NiSi films [2]. The addition of a small amount of Pt also increases the temperature of agglomeration of NiSi [3]. Ni(Pt)Si thin film alloys are thus currently used as contact layers in complementary metal–oxide-semiconductors (CMOS) to improve the integration of these devices in nanometric transistors [4]. Extensive work has also been done on determining the effects of alloying element on the formation and stability of Ni silicides [5,6]. The reactions of pure Ni thin films with Si are characterized by the sequential growth of Ni_2Si and NiSi [7,8]. Their growth is limited by grain boundary diffusion of Ni, which is the dominant diffusing species in these silicides [9]. Recently, more complex growth behavior [10] including the formation of "transient phases", has been observed. The presence of Pt in Ni film suppresses the "transient" Ni-rich phases. The simultaneous growth of NiSi and Ni_2Si during the consumption of the layer Ni alloyed with Pt has been observed by atom probe tomography (APT) [11], X-ray diffraction and transmission electron microscopy (TEM) [12]. However, the fundamental mechanisms behind these effects are not fully understood.

It has been shown that silicide formation is accompanied by stress and concomitant relaxation of this stress. In particular, the Ni_2Si phase formation induces a compressive intrinsic stress that relaxes after the complete formation of Ni_2Si phase in the case of a pure nickel film [13]. Recently, we have observed that the growth of NiSi starts (in the pure Ni case) or is accelerated (Ni(Pt) case) when the stress in Ni_2Si is relaxed but not when the growth of Ni_2Si is completed [14]. It was also shown that the relaxation is slower in the presence of Pt compared to the pure Ni case.

The redistribution of Pt may also affect silicide formation. Atom probe tomography analysis has shown that the redistribution of Pt at Ni(Pt)/Ni_2Si interface during the Ni silicide formation is characterized by the "snowplow" effect [11].

In this paper, a Ni silicide alloy with Pt annealed at low temperature was characterized by APT [15]. Three-dimensional (3D) composition mapping and the morphological information of the different silicides are determined. The redistribution of Pt in Ni silicides is

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analyzed in correlation with the formation and the morphology of the different phases.

Fifty nanometers of Ni(5% Pt) films were prepared by sputtering of a Ni(Pt) alloy target with 5 at.% Pt. The Si(100) substrates were cleaned with dilute HF prior to loading into the sputtering chamber. In situ isothermal X-ray diffraction at 300 °C was performed on Ni(5% Pt)/(100)Si and the experiment was stopped before the Ni(Pt) layer was totally consumed (i.e., after 1 h). Simultaneous formation of NiSi and Ni_2Si was observed [12].

Specimens for 3D APT were prepared from blanket structures using the lift-out technique [16]. Atom probe specimen preparation was conducted in two different orientations with the analysis direction either parallel or perpendicular to the growth. In this study, data from the former specimen orientation are presented. The tips were prepared using the annular milling method [17] to achieve an end radius of ~100 nm. Pulsed picosecond-laser APT was performed for compositional profiling of the thin film with subnanometer scale spatial resolution [18]. Analyses were performed with an Imago Scientific Instruments LEAP 3000X HR at a specimen temperature of 50 K, a 200 kHz pulse repetition rate, an energy of 0.6 nJ $pulse^{-1}$, an evaporation rate of 0.01 ions $pulse^{-1}$ and a pressure of $<3 \times 10^{-9}$ Pa.

An example of large field of view APT analysis is shown in Figure 1 where Ni and Si atoms and Pt atoms are shown (Fig. 1a and b, respectively). This large volume for an atom probe measurement allows statistical information at the atomic scale to be obtained from one dataset. In addition to the Si substrate, three regions have been identified based on their Ni composition (i) unreacted Ni(Pt) film; (ii) Ni_2Si phase with a thickness of approximately 60 nm; (iii) a non-continuous layer containing NiSi grains. These regions are represented schematically in Figure 1c. Considerable interface roughness is observed and in particular the interface between the Ni_2Si film and the Si substrate is very rough. The NiSi phase was found to form large islands at the Ni_2Si/Si interface.

To better understand the morphology of the phases and the Pt distribution therein, the volumes of the NiSi and Ni_2Si phases were determined by using the following concentration range: 45–55 at.% Ni for NiSi and 60–70 at.% Ni for Ni_2Si. Figure 1c shows the location of these phases and Figure 2b shows the NiSi phase volume viewed from the top of the layer. The NiSi phase forms a discontinuous layer at the Ni_2Si/Si interface with a thickness of the order of 15 nm. The NiSi precipitates have complex shapes and different sizes. The empty regions in this volume represent regions containing the Ni_2Si/Si interface.

For the Ni_2Si layer, it was found that Pt can be used to reveal the grains boundaries as shown in Figure 2a, which represents a top view of the 3D reconstruction volume of the Ni_2Si phase. To obtain this view, a 20 nm slice was taken in the Ni_2Si phase and only the Pt atoms corresponding to a concentration higher than 1.5 at.% were plotted. It can be seen that the Pt atoms are preferentially present at the grain boundaries, acting as markers to identify grains of the Ni_2Si phase. The grain structure has a classical columnar structure with an average grain size

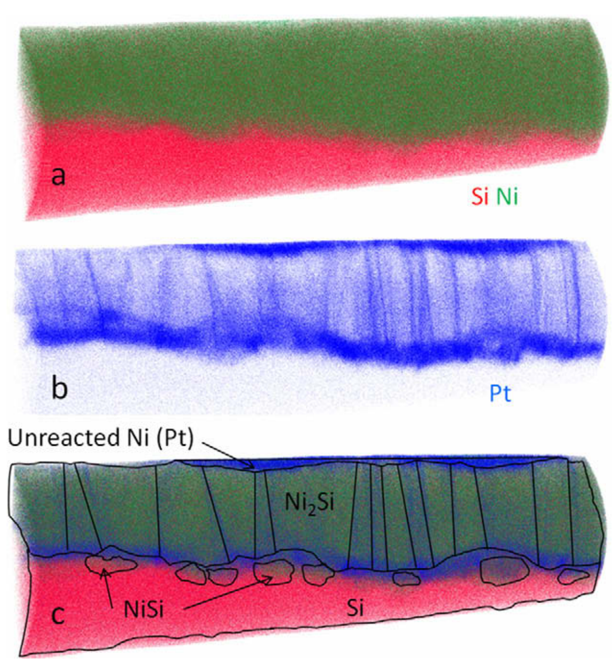

Figure 1. The APT reconstruction of a large ($120 \times 120 \times 400$ nm^3) analysis volume collected from the Ni silicide structure. (a) Si and Ni atoms. (b) The location of the Pt atoms. Pt enrichment is observed at the top of the structure (corresponding to the unreacted Ni layer) and at the interface between silicide and silicon regions. Note the large roughness present at this lower interface. A schematic of the structure is also shown in (c), which highlights the location and morphology of both silicide phases detected.

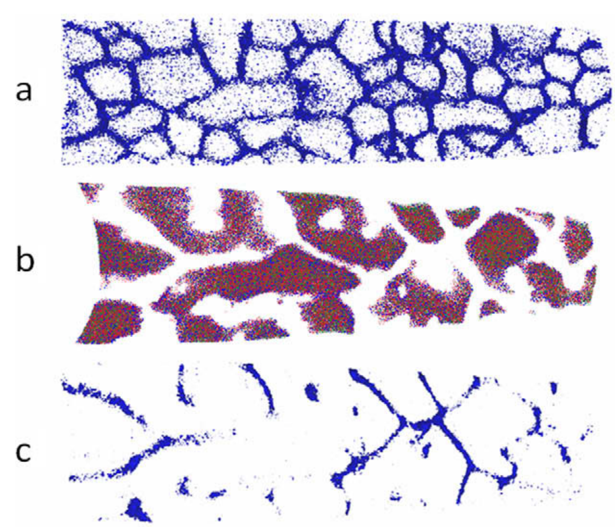

Figure 2. The Pt distribution within both Ni_2Si and NiSi regions (thin slice $\times$ 120 $\times$ 400 nm^3). (a) A 20 nm slice in depth through the Ni_2Si region; significant grain boundary enrichment of Pt (2 at.% Pt isoconcentration surfaces shown) can be seen. In one single APT analysis it is now possible to capture many (~50) of these columnar features. Regions of NiSi were also observed and the morphology of the NiSi grains are shown in (b). The NiSi layer is clearly seen to be discontinuous, and examination of the Pt distribution close to the NiSi (c) (40 nm slice) shows that high levels of Pt are present in the regions between the NiSi grains. In this case 8 at.% Pt isoconcentration surfaces are shown.