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An investigation into interfacial oxide in direct silicon bonding

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ABSTRACT

In this work we have used transmission electron microscopy (TEM) to image the joined interface for hydrophobic direct silicon to silicon fusion bonding. The factors investigated included annealing temperature, oxygen content in the silicon, crystal orientation and doping level. Samples were prepared by joining polished silicon device wafers to polished silicon handles. The joined pairs were annealed at temperatures between 600°C and 1200°C, and the device wafers thinned by grinding and polishing. The interfaces were analyzed for interfacial oxide and crystalline defects using a high resolution TEM. Secondary ion mass spectroscopy was used to analyze the oxygen and dopant profiles along the interface. The electrical characteristics and movement of impurities across the bonded interface were investigated by spreading resistance profiling.

INTRODUCTION

Silicon–silicon bonding has its application in the manufacture of PIN diodes and IGBT transistors, using layers of single crystal silicon with different electrical characteristics. Deposited silicon (epitaxy) is currently used to make silicon layers of differing resistivity; however single crystal silicon has fewer defects, while thick epitaxial silicon can be expensive to produce. Silicon to silicon bonding allows layers of different electrical characteristics to be produced in one substrate, increasing the reliability of the product. Epitaxy also requires a thermal cycle for deposition, which can make the production of diodes without an interfacial oxide difficult (1).

The interfacial oxide plays an important part in device characteristics and acts as a barrier to dopant movement across the bonded interface. The base current of polysilicon emitter bipolar transistors is reduced by the presence of oxide at the polysilicon interface, which creates a barrier to electron flow and gives additional capacitance. However, the presence of oxide also increases the gain, and annealing is required to optimise the level of contiguous oxide present. For IGBT's, the interfacial oxide acts as a barrier to electron flow and therefore needs to be minimized.

In this paper, direct silicon bonding with very low levels of interfacial oxide is presented. The mechanisms controlling the presence of oxide and the characteristics of the oxide after annealing are investigated. The interfacial oxide originates from the native oxide and its thickness is controlled by the bonding environment and pre-bond cleaning process. The interfacial oxide is trapped at the joined interface and begins to migrate to form islands at above 1100°C (2). Migration of the interfacial oxide as a function of

temperature, and the formation of small oxide islands at the joined interface under high temperature annealing will be described. The non-symmetrical formation of oxide islands as a function of doping concentration and material type is also presented, as well as the effect of oxygen content in Czochralski (Cz) and float-zone (FZ) material.

EXPERIMENTAL

This report covers material differences of growth, orientation, and dopant, and correlates the effects with anneal temperature. Six different types of material were used, as shown in Table 1, labelled A-F. The handle wafers were prepared for bonding by using chemical-mechanical polishing to achieve a low surface roughness and good thickness variation. The wafers were cleaned pre-bond with a dilute HF process to minimize the native oxide and provide a hydrophobic surface for the silicon to silicon bond. The wafers were joined within 60 sec of completion of the drying cycle, and all were joined simultaneously. A high resistivity, n-type, FZ, <111> device (group A) was bonded to each type listed, and all samples were immediately annealed in nitrogen at 600°C. This was to ensure that no out-gassing occured to affect the results between joining and annealing. Subsequent annealing at temperatures of 1000°C, 1050°C, 1100°C, 1150°C and 1200°C were performed in nitrogen on each of the six groups. The device wafers were then ground and polished back to 2 μm for high resolution secondary ion mass spectroscopy (SIMS) and transmission electron microscopy (TEM). The samples were prepared for TEM by mechanical polishing and Ar^+ ion milling from the centre of the wafer. The analysis was performed in a JEOL 4000EX TEM at 400 keV. All TEM images shown are bright field. The SIMS profiles were obtained using a Cameca 4f spectrometer with a Cs^+ primary ion beam. Quantitation was obtained by reference to implant standards in silicon.

RESULTS AND DISCUSSION

Figure 1 shows a TEM image of an FZ device material bonded to group E, FZ, n-type, 14-20 ohm.cm handle material after annealing at 1150°C in nitrogen. No interfacial oxide is observed along the bonding interface. The TEM investigations indicate that similarly no oxide islands are observed in the case of group A, FZ, n-type material bonded to a similar n^-, FZ device. Thus, in the case of FZ material, the native oxide formed prior to bonding, which is a few monolayers thick, appears to be absorbed into the bulk region at temperatures of 1150°C and above.

Figure 2 indicates a TEM image of an oxide island formed after bonding n-type, Cz material of 6-10 ohm.cm resistivity to an n^-, FZ device and annealing at 1200°C. The image clearly shows an asymmetrical oxide island formed predominantly in the Cz silicon. The oxygen concentration in the Cz material has been measured to be 2-7 x 10^{17} atoms/cm^3 using high resolution SIMS, which is above the limit of solid solubility of oxygen in silicon at 1000°C. The limit of solid solubility increases to 7 x 10^{17} atoms/cm^3 at 1200°C for oxygen in silicon. At these levels the oxygen in the bulk is at or near saturation, and diffuses to the oxide island, increasing the volume of the oxide at the interface. The interfacial oxygen diffuses along the interface to form islands, and some

interstitial oxygen diffusing across the interface can be expected to follow the same diffusion path to the oxide island.

The dopant concentration also plays a role in oxide island formation, increasing the volume of the islands formed in highly doped substrates. Figure 3 illustrates an oxide island formed in a sample from group B, showing an almost symmetrical nature due to the diffusion of arsenic and oxygen into the high-resistivity FZ device layer at 1200^oC, with subsequent silicon dioxide growth in that layer. Similar formations have been observed with highly doped n^+ handle material annealed at 1150^oC. The diffusion of arsenic and oxygen into the device material has been characterized by SIMS, as shown in Figure 4. This indicates the diffusion of arsenic and oxygen to a depth of several microns into the device. In this experiment, the area of oxide islands imaged under TEM for groups B & C was three times larger than comparable material of lower doping level (group D). Similar results were found for highly doped antimony substrates (group F). Since previous work has shown that the oxidation rate is enhanced in highly doped n^+ silicon (4) resulting in larger oxide island formation, the results imply that the mechanism for oxide island growth here might be similar to thermal oxidation rather than precipitate formation, because the latter can be suppressed by a high arsenic concentration (5).

Dislocations were visible when material of the same orientation was bonded, but were greatly reduced when bonding different orientations. The formation of oxide islands occurred to the same extent in all Cz material regardless of orientation. Plan view TEM imaging was used to examine the level of oxide island formation and the occurrence of dislocations. Figure 5 depicts a plan view TEM of oxide islands. The observed finely spaced Moire fringes have a spacing of 7.1 nm, indicating a rotation between common [022] type directions of 1.55^o. Larger Moire fringes have a spacing of 40.4 nm and their origin is unknown. The oxide islands switch from black to white contrast due to bending of the sample. Oxide islands can be seen to form in bands similar to oxygen precipitation formation in Cz material. Figure 6 is a plan view TEM of <111> n^+ material (group C) bonded to an n^-, <111>, FZ device material. The observed Moire fringes have a spacing of 4.6 nm, corresponding to a rotation of the two lattices by 2.4^o. This rotation results in dislocations, while oxide islands cannot be clearly observed. Further TEM investigations of dislocations have indicated that no correlation with oxide island formation is evident.

The samples were initially annealed at 600^oC to minimize the effect of the environment on the interfacial oxide. However, voids are readily formed at 600^oC from out-gassing unless the final rinse step is omitted. For safety reasons this is not a procedure used for this work, and the voids formed were eliminated after annealing at above 900^oC. This procedure resulted in a native oxide a few monolayers thick, and enabled dissolution of the native oxide in FZ material. It has been reported previously that dissolution of the native oxide is not likely for thicker native oxides of 10 **Å** or more (3). It is clearly visible from TEM imaging (Figure 1) that dissolution occurs due to the solid solubility of oxygen in silicon of 5×10^{17} atoms/cm^3 at 1150^oC. The high resolution TEM image of Figure 7 shows a crystalline bond without misfit dislocations achieved by bonding <111> orientation to <100> orientation.

CONCLUSIONS

The results from this work indicate that the initial formation of oxide islands is due to diffusion along the silicon bonded interface to energetically favourable oxide island sites. In float-zone silicon the oxide islands subsequently diffuse into the silicon

during the annealing process, due to the solid solubility of oxygen in silicon. The oxide island growth is enhanced by the supersaturation of oxygen in Cz silicon, clearly indicated by the asymmetrical oxide formation at the Cz/FZ interface. Oxide island formation is further enhanced in highly doped n-type material. This indicates that the oxide island growth mechanism in Cz material is similar to the growth of thermal oxide on silicon in an oxygen ambient. The oxide islands do not tend to favour dislocation sites, and their occurrence appears to be dependent on the initial movement of the interfacial oxide and subsequent growth at certain sites along the interface.

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Group	Diameter	Type	Dopant	Resistivity	Orientation	Growth
A	100 mm	n^-	Phosphorous	2-4 kohmcm	<111>	FZ
B	100 mm	n^+	Arsenic	0-0.07 ohmcm	<100>	Cz
C	100 mm	n^+	Arsenic	0-0.07 ohmcm	<111>	Cz
D	100 mm	n	Phosphorous	6-10 ohmcm	<100>	Cz
E	100 mm	n	Phosphorous	14-20 ohmcm	<100>	FZ
F	100 mm	n^+	Antimony	0.01 ohmcm	<100>	Cz

Table 1. Material properties.

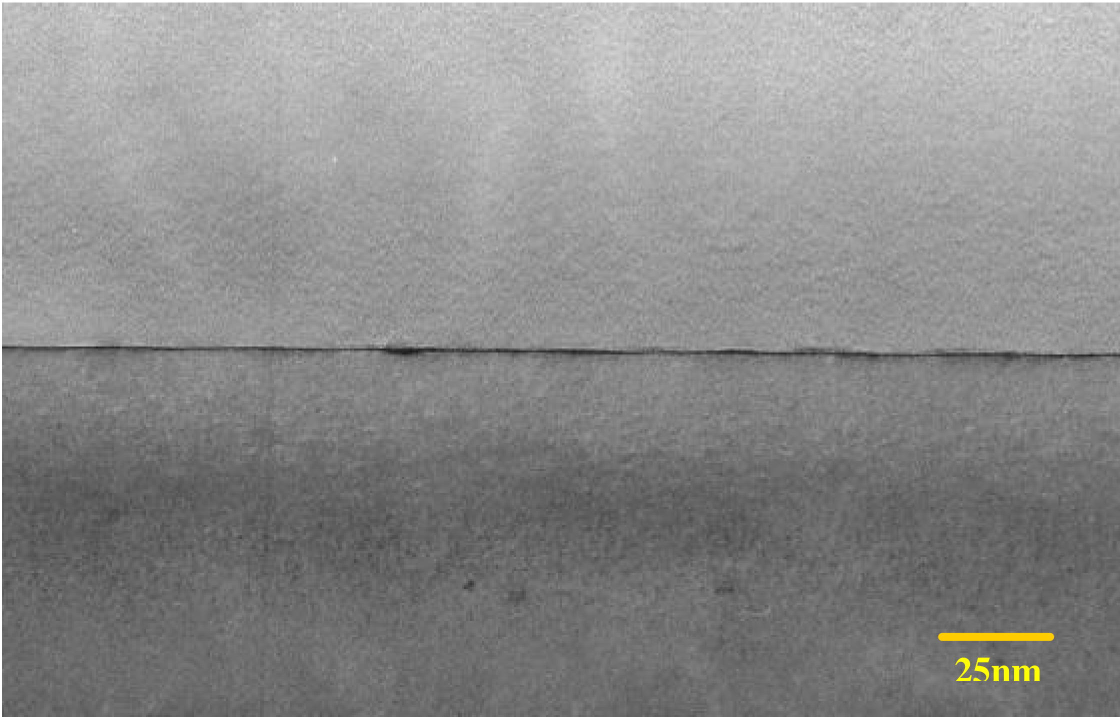

Figure 1. TEM indicating no oxide island formation with FZ (Group E).

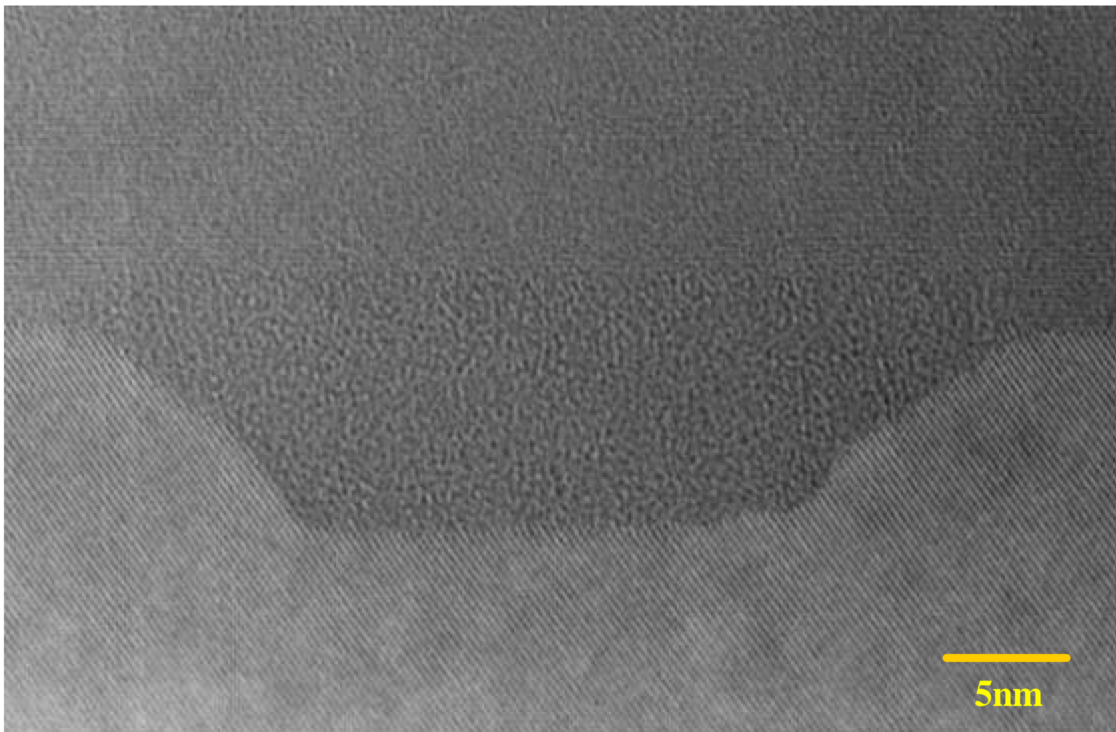

Figure 2. Asymmetrical oxide island formation in Cz directly bonded to FZ material.

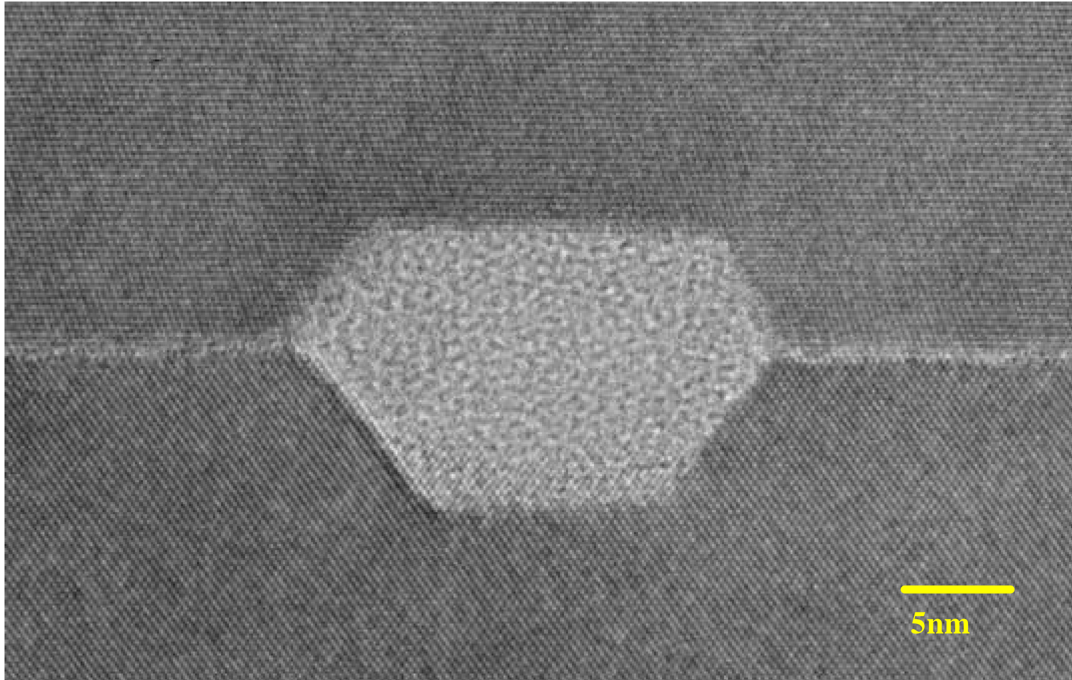

Figure 3. Oxide island formation in highly doped material (Group B).

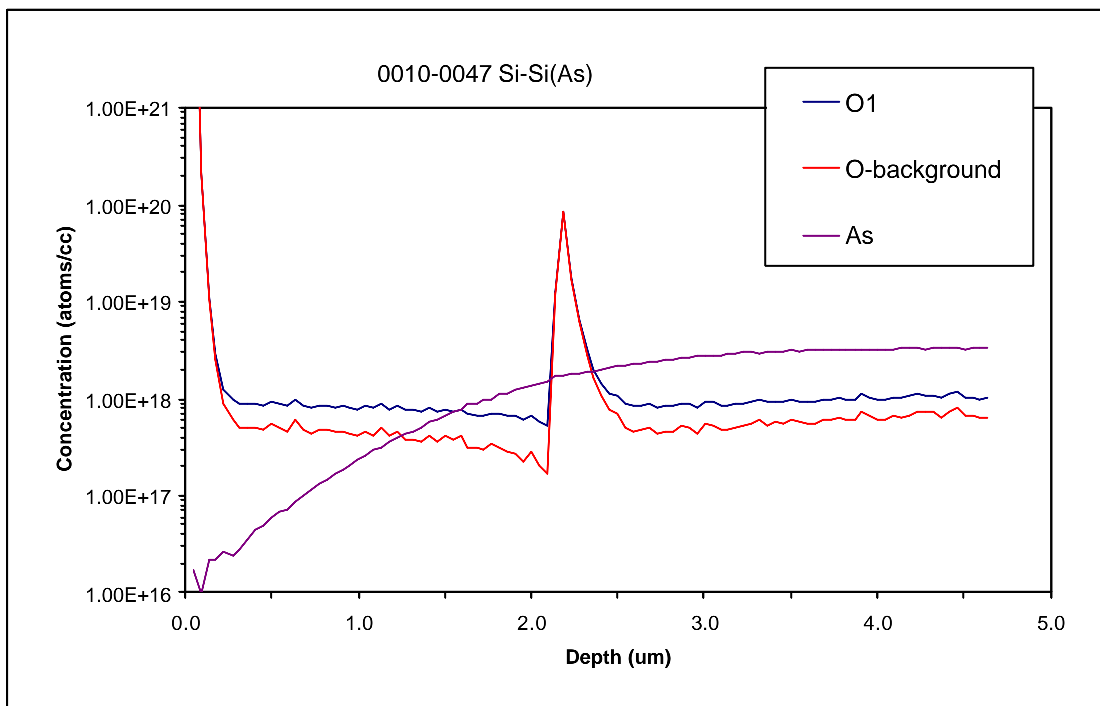

Figure 4. Arsenic and oxygen SIMS of a silicon to silicon fusion bonded interface.

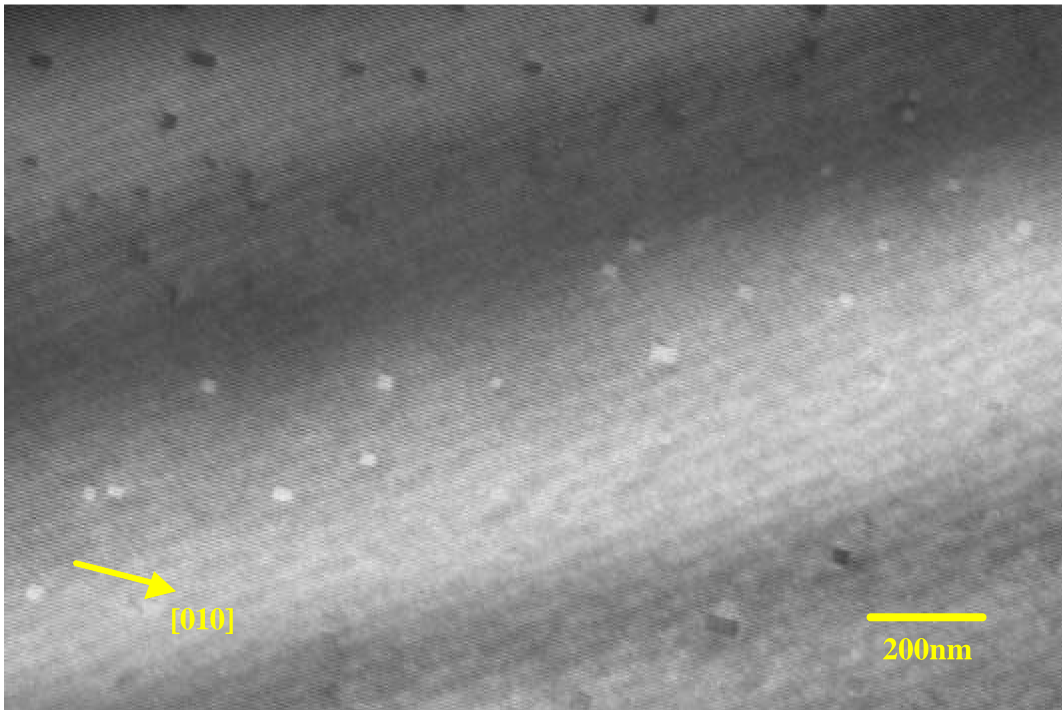

Figure 5. Plan view TEM (Group B) indicating oxide islands.

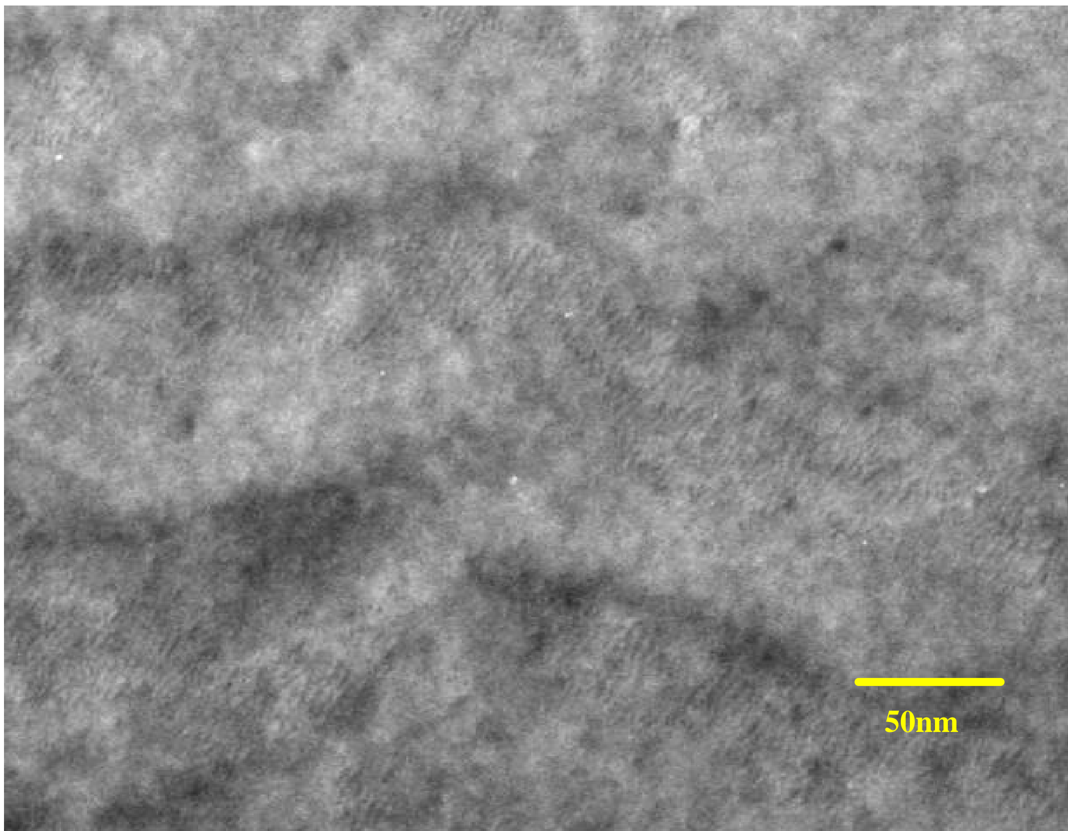

Figure 6. Plan view TEM (Group C) indicating Moire fringes and precipitates.

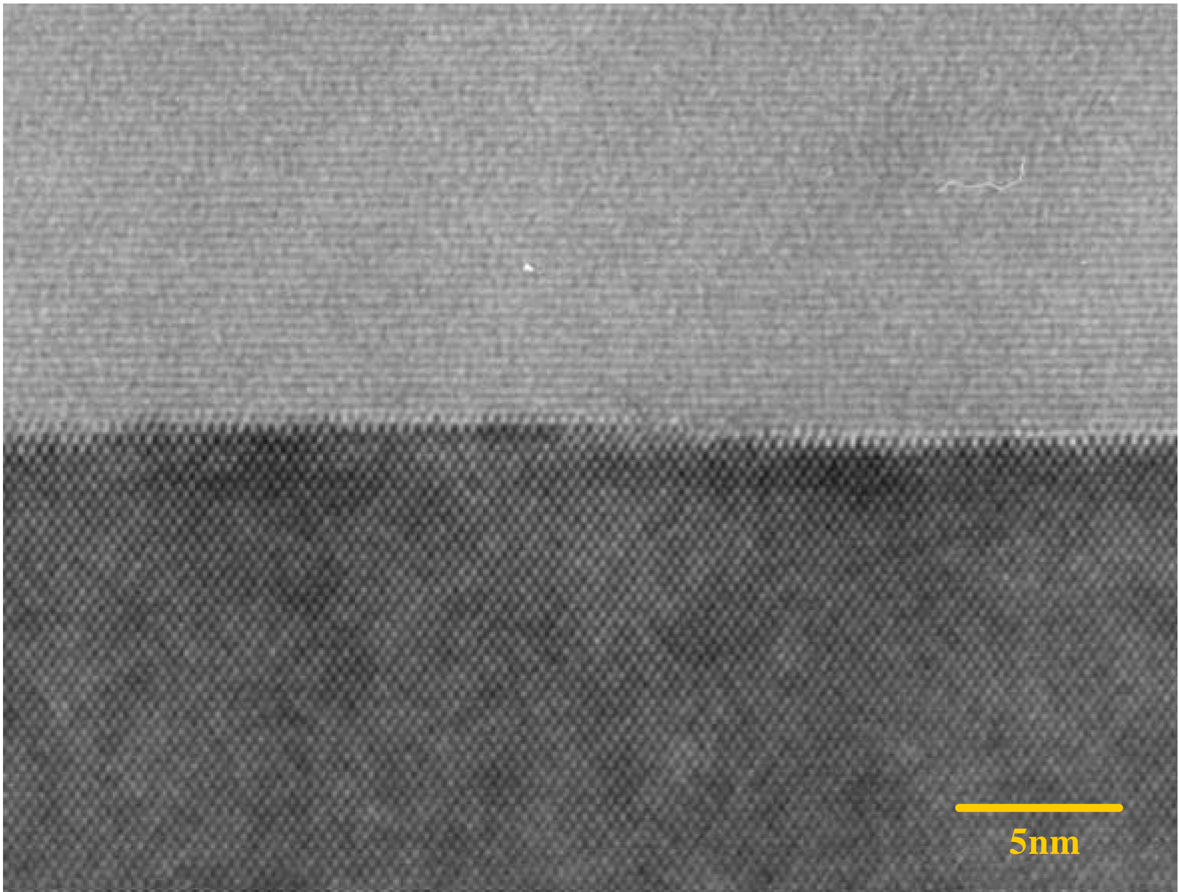

Figure 7. High resolution TEM showing a crystalline <100> to <111> orientation silicon to silicon bond.