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Formation of SiC layer on Single Crystal Si Using Hot-Filament Reactor

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The effect of gas activation on the formation of SiC layer on Si substrate using methane as a carbon source was investigated. Tungsten filaments, heated above 2000°C, were used to activate the methane-hydrogen mixed gas. The dissociation of methane gas by the heated filament was enough to form a SiC layer successfully, which was very difficult without any activation. The SiC layer formed on the Si substrate was crystalline and nearly epitaxial as measured by X-ray diffraction. The stoichiometry was also close to 1:1. However, the characteristic of the SiC layer was dependent on the heat-treatment condition. The general behavior of the layer growth with the variables was discussed.

Key words : SiC, Methane precursor, Gas activation, Hot filament, Epitaxial layer

I. Introduction

SiC is one of the candidate materials for high temperature or high speed semiconductor device. Although single crystal SiC wafers are being produced, the price is very high and the size is limited. The epitaxial growth of SiC on Si wafer is, thus, very important in general uses for semiconductor application.[1-3] Two kinds of trials have been done to form a SiC epitaxial layer on Si. One is to use a carbonization reaction between carbon containing gas species and a Si substrate.[4-9] Carbon species react with Si atoms on the substrate surface and SiC layer grows by consuming the Si atoms of the substrate. No Si atom sources except the substrate is needed. Another is to use the CVD reaction between carbon containing and Si containing gas species on the substrate surface [10-14] However, even in the CVD process, the carbonization process is necessary to grow a perfect SiC epitaxial layer.

Several methods have been tried for the carbonization processes.[5,8,9,15] A typical one among them is a thermal process : the Si substrate is treated at high temperature under the carbon containing atmosphere. No activation of gas other than substrate heating is applied. The gas used, thus, should be large hydrocarbon molecules such as propane in order to make the dissociation easy. Methane is not used because of the difficulty of dissociation, but it is easy to purchase and inhibit the soot formation. In this paper, we investigated the possibility of carbonization process using methane by applying an activation of gas by thermal energy.

II. Experimental Procedure

The reactor was very similar to that used for diamond CVD. Eight tungsten filaments were installed parallel one another above the substrate. The length of the substrate and filament was 5 mm. The filaments were heated above 2000°C, which had a role both of activating the gas and heating the substrate. The Substrate temperature was controlled between 1000°C and 1300°C by controlling the filament temperature. Total flow rate of CH_4-H_2 mixed gas was 100 sccm, of which CH_4 was varied from 0 5 to 6%. After raising the pressure to 40 torr, the filament was heated to a set temperature for 30 min. Then, the substrate was put on the exact position and the reaction started. (100) Si substrate was used with the size of 3×3 cm^2. It was cleaned with acetone, alcohol and then etched with 5% HF solution for 3 min. It was cleansed with distilled water and blown with dry nitrogen.

III. Results and Discussion

Since the reactor and its treatment condition are very similar to those used in diamond CVD,[16] it is possible for the diamond to deposit on the substrate. In this case, however, the diamond was not observed on the substrate even after a long treatment. This is because the substrate was used without any nucleation enhancement treatment for diamond nucleation and treatment temperatures were higher than that proper to diamond deposition.

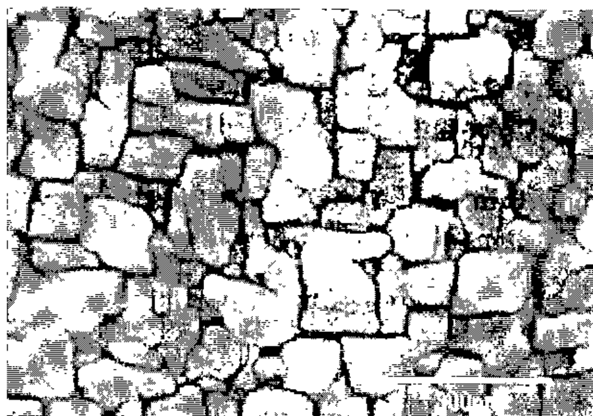

Fig. 1 Surface morphology of Si substrate treated at 1300°C with 3% CH_4 for 5hrs

The typical surface morphology of the substrate after the treatment is shown ing Fig. 1. Faceted grains form on the substrate, who look aligned parallel one another. This is very similar to those reported previously[17] using hydrocaron gases larger that CH_4, which implies the gas activation is efficient in making gas species incorporating SiC formation.

The crystal structure of the film was examined by X-ray diffraction (XRD), Raman spectroscopy and its stoichiometry by Auger electron spectrum (AES). Fig. 2 shows a Raman spectrum. The peak is not sharp but broadened. The wavenumber of β-SiC is 970 cm^{1}. Whether the broadening is caused by the mixture with other peaks or the thinness of the layer is not clear. Fig. 3 shows a XRD of the layer formed on the substrate. Only (200) SiC peak is observed on the spectrum. This indicates the SiC layer is crystalline and the grains have their <200> orientation normal to the substrate. To clarify if this film is epitaxial to the substrate, we measured a pole figure. Fig. 4 shows a pole figure of the layer. It shows that the layer has a four fold symmetry. It is, thus, condluded that the layer has an epitaxial relation with the substrate.

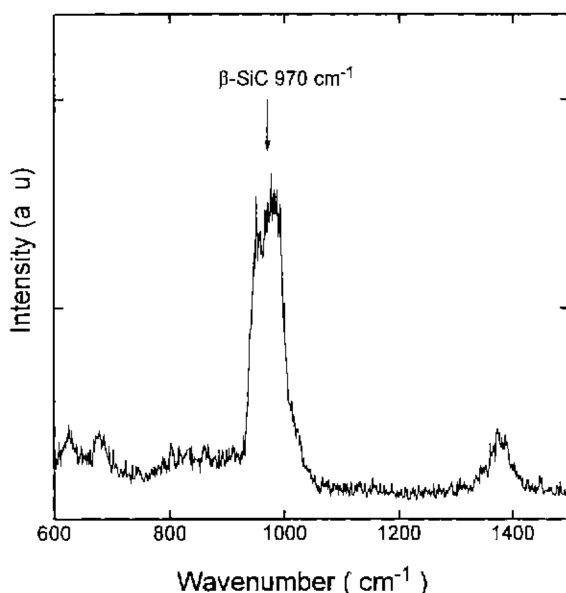

Fig. 2 Raman spectrum of the specimen treated at 1300°C with 3% CH_4 for 5 hrs.

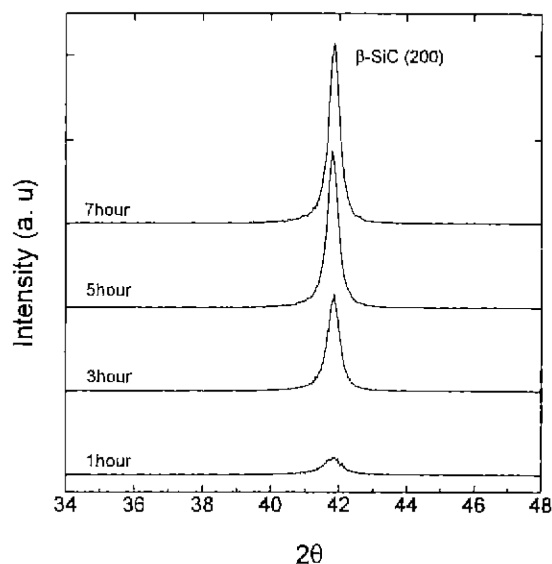

Fig. 3. X-ray diffraction patterns of the specimen treated at 1300°C with 3% CH_4 for various time

Fig. 5 shows the depth profile of the ratio of carbon to silicon. The region showing a plateau, which is considered as a stoichiometric SiC layer. The carbon to silicon ratio is nearly 1 in the case of treating at 1300°C, but less smaller at 1250°C.

The growth behavior of the layer such as the grain size, thickness, stoichiometry, the degree of epitaxy varied with the treatment condition. The treatment tem-

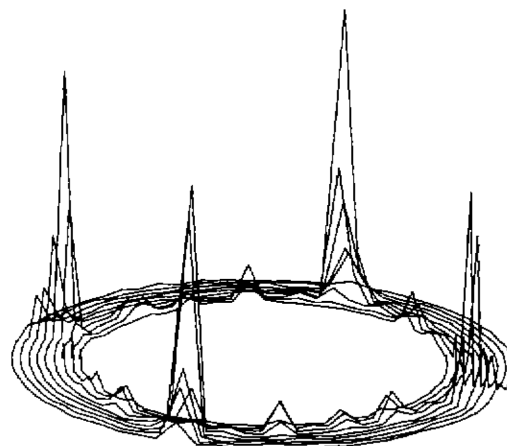

Fig. 4. {111} pole figure of the specimen treated at 1300°C with 3% CH_4 for 5hrs.

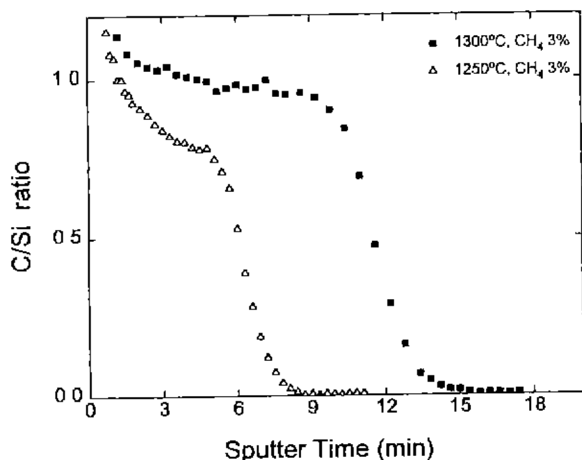

Fig. 5. AES depth profiles of the specimens treated at 1250°C and 1300°C for 5hrs respectively.

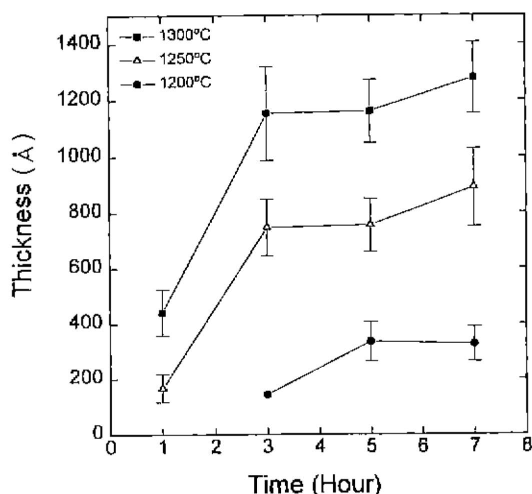

Fig. 6. Thickness variation of SiC layer with treatment time with 3% CH_4 at various temperature.

perature and the methane concentration appeared as the most important parameters. The growth of the layer was saturated with the treatment time. Fig. 6 shows the thickness variation of the layer with the treatment time at various temperatures. The thickness is nearly saturated after 3 hrs of treatment and the saturated thickness increases with the temperature. The grain size on the surface showed the variation similar to that of the thickness. Consequently, the grain size increased as the thickness increased. The degree of alignment can be estimated by comparing the full width at half maximum (FWHM) of the peak measured in the pole figure. By comparing these values, the degree of the alignment was shown to increase as the treatment temperature and treatment time became higher.

IV. Conclusions

The activation of gas was verified very effective enough to form SiC layer on the Si substrate. Even methane gas formed SiC layer so successfully even at the temperature as low as that used by researchers using large molecular hydrocarbons previously. Accordingly the activation of gas can make the treatment temperature much lower, which is important in application.

V. Acknowledgement

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