

## Phase Distribution and Interface Chemistry by Solid State SiC/Ni Reaction

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The phase distribution and interface chemistry by the solid-state reaction between SiC and nickel were studied at temperatures between 550 and 1250°C for 0.5-100 h. The reaction with the formation of silicides and carbon was first observed above 650°C. At 750°C, as the reaction proceeded, the initially formed Ni<sub>3</sub>Si<sub>2</sub> layer was converted to Ni<sub>2</sub>Si. The thin nickel film reacted completely with SiC after annealing at 950°C for 2 h. The thermodynamically stable Ni<sub>2</sub>Si is the only observed silicide in the reaction zone up to 1050°C. The formation of Ni<sub>2</sub>Si layers with carbon precipitates alternated periodically with the carbon free layers. At temperatures between 950°C and 1050°C, the typical layer sequence in the reaction zone is determined by quantitative microanalysis to be SiC/Ni<sub>2</sub>Si+C/Ni<sub>2</sub>Si/Ni<sub>2</sub>Si+C/Ni<sub>2</sub>Si/Ni(Si)/Ni. The mechanism of the periodic band structure formation with the carbon precipitation behaviour was discussed in terms of reaction kinetics and thermodynamic considerations. The reaction kinetics is proposed to estimate the effective reaction constant from the parabolic growth of the reaction zone.

**Key words :** Silicon carbide, Nickel, Periodic band structure, Reaction kinetics, Thermodynamic driving force

### I. Introduction

For many applications it is important that the fundamentals of phase distribution and interface chemistry by solid state reactions between ceramics and metals are fully understood. Recently, several studies have been reported to develop some basic understanding on the physical and chemical interactions at the SiC-metal interfaces<sup>1-3)</sup>.

Nickel could be used to make good high-temperature contacts for utilizing the properties of SiC. It is also of specific interest due to its silicide-forming tendency at low temperature, making a suitable material for metallization. A few detailed studies of the interaction of thin nickel films with SiC at low temperatures have been reported<sup>4-6)</sup>. In previous studies<sup>7,8)</sup>, the interaction between SiC and cobalt films and foils in the temperature range between 500 and 1450°C were reported. These investigations aimed at providing the surface morphology, reaction products and interface structure.

In this paper, the phase distribution and interface chemistry by the solid state SiC/Ni reaction in the temperature range between 550 and 1250°C were studied. The reaction products are presented using thin nickel films deposited on SiC substrates. More detailed informations about the formed interface like periodic band structure and carbon precipitation are investigated using thick nickel foils.

### II. Experimental Procedure

The materials used for the experiments were highly dense sintered  $\alpha$ -SiC form "Elektroschmelzwerk Kempten", ESK, and thin sputter nickel films as well as thick nickel foils from Alfa Prod./Johnson Matthey Company. The polycrystalline SiC contained 1.5 wt% total impurities, such as carbon and aluminium (ESK). The SiC plates were cut into small pieces with a diameter of 20 mm and a thickness of 3 mm and ground with a BN/C disk. The ground SiC samples were then polished using diamond pastes of 30, 15, 3, 1  $\mu$ m. Nickel films were sputter-deposited on to the polished SiC substrates in a commercial sputter equipment (Leybold/Germany, type Z-400). A magnetron-type D.C. sputtering source was used to deposit the coatings. The growth rate of nickel films was calculated to be 667 nm/min under the conditions used (D.C. potential 400 V, partial pressure of Ar  $7.0 \times 10^{-2}$  mbar and bias voltage -150 V). The applied film thickness was fixed at 2.0  $\mu$ m for this study. This thickness allows the penetration of the CuK $\alpha$  beam entirely into the reaction zone by X-ray diffraction analysis (XRD). The nickel foil has a purity of 99.997% and a thickness of about 0.5 to 1 mm.

The couples of SiC with thin sputtered nickel films were annealed at temperatures between 550 and 1250°C for 0.5-2 h, and the couples of SiC with thick nickel foils at temperatures between 950 and 1250°C for 4-100 h. These annealing experiments were conducted in a high-

temperature vacuum furnace manufactured by Degussa/Germany. SiC/Ni samples were placed in a graphite crucible. The specimens were surrounded by titanium to remove the residual oxygen during the annealing time. After positioning the samples, the furnace was evacuated to  $6 \times 10^{-6}$  mbar and subsequently filled with a gas mixture of Ar/4 vol% H<sub>2</sub> for the annealing time. Thermocouples of type EL18(PtRh30/PtRh6) were used for temperature measurement. The heating rate was set between 20 and 30 Kmin<sup>-1</sup> and the cooling rate between 5 and 10 Kmin<sup>-1</sup>.

For the prediction of all reaction products, the qualitative process of the reaction in SiC with thin sputtered nickel films was estimated by determining phase fractions from relative XRD peak intensities of at least three coincidence-free reflections. The reaction couples of SiC with thick nickel foils were cut by a diamond saw and then embedded in copper resin. After mounting the reaction couples were ground on a diamond disk, polished with diamond pastes of 30, 15, 6, 3 and 1  $\mu\text{m}$ . The polished cross-sections were investigated using optical microscopy, scanning electron microscopy (SEM). Quantitative atomic concentration profiles of silicon, carbon and nickel were measured using electron probe microanalysis (EPMA).

### III. Results

#### 1. Reaction products

The reaction products were identified by determining the relative XRD peak intensities. Fig. 1 shows the qualitative phase analysis of the reaction couples of SiC with thin sputtered nickel films based on the XRD analysis for different temperatures and times. Below 650°C, no reaction was observed. At 650°C, the nickel layer reacted and formed the silicides Ni<sub>2</sub>Si and Ni<sub>3</sub>Si<sub>2</sub>. Unreacted nickel was distributed on the film surface. No carbide formation, either Ni<sub>2</sub>C or Ni<sub>3</sub>C, was detected by XRD. At 750°C, the Ni<sub>3</sub>Si<sub>2</sub> phase was transformed to be the Ni<sub>2</sub>Si

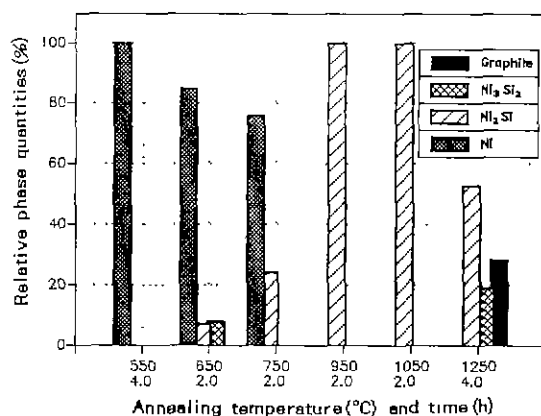


Fig. 1. Qualitative analysis of the reaction products between SiC and thin sputtered nickel films based on relative XRD phase estimation for various annealing conditions.

phase. At 950°C the entire nickel films were fully consumed after 2 h. The Ni<sub>2</sub>Si is the only observed silicide in the reaction zone up to 1050°C. After 2 h at 1250°C, the Ni<sub>2</sub>Si phase with small quantities of Ni<sub>3</sub>Si<sub>2</sub> and graphite were detected. The quantity of graphite could be detected by the graphitization of carbon.

The reason why the Ni<sub>3</sub>Si<sub>2</sub> phase was formed at 1250°C may be attributed to the fully consumed nickel and the predominant diffusion of silicon in the reaction zone. Because of the limited thickness of the thin nickel films following exposure for a few hours at elevated temperature, the complete reaction path about the formed interface like periodic band structure and carbon precipitation could be determined in the following investigation using thick nickel foils.

#### 2. Phase distribution

In this section, since the reaction behaviour at 950°C is similar to that at 1050°C, the emphasis is on the results obtained at 950°C for 100 h. Fig. 2 shows a cross-sectional view of the overall reaction zone of SiC with thick nickel foils after annealing at 950°C for 100 h. The reaction zone consists of various kinds of distinct regions. This constitution could be identified by measurements using EPMA. Based on EPMA, quantitative analysis over the overall reaction zones is presented in the form of line scans for the elements silicon, carbon and nickel in Fig. 3, which corresponds to the whole region in Fig. 2. Adjacent to the SiC a large zone can be recognized with alternating layers (Fig. 2(b)). The carbon intensity alternates in this region corresponding to the bright and dark alternating layer sequence in Fig. 3(b).

The average concentration of the SiC and the bright and dark alternating layers is summarized in Table 1. The average concentration of nickel, silicon and carbon in the dark layer shows 64.6 wt% nickel, 16.8 wt% silicon and 18.6 wt% carbon corresponding to the value of 33.9 at% nickel, 18.4 at% silicon and 47.7 at% carbon. In the

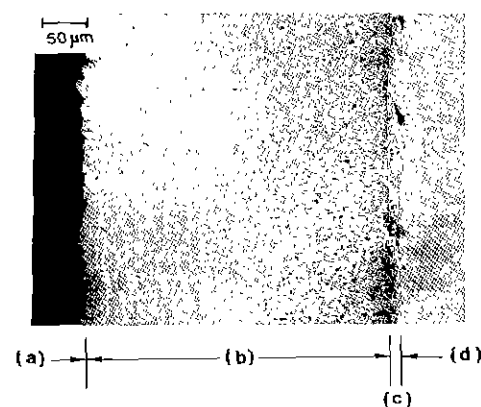
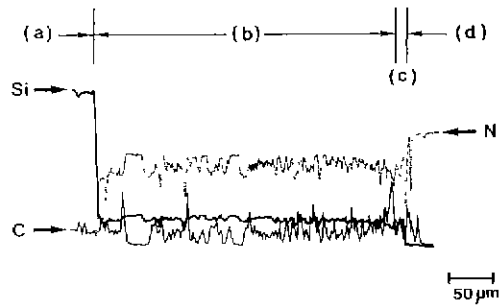
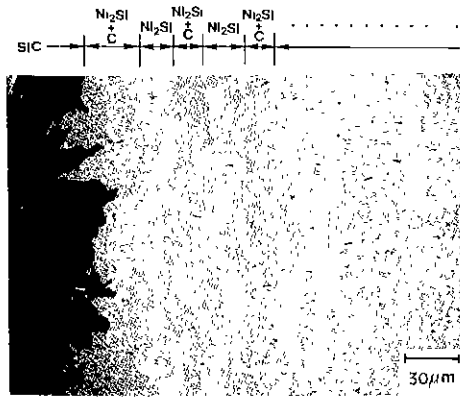


Fig. 2. SEM micrograph of the cross-sectional view of SiC/Ni after annealing at 950°C for 100 h; (a) SiC, (b) SiC reaction zone with alternating layers of Ni<sub>3</sub>Si<sub>2</sub>/Ni<sub>2</sub>Si, (c) metal reaction zone of Ni<sub>2</sub>Si and (d) nickel metal.



**Fig. 3.** EPMA line scan for silicon, nickel and carbon over the corresponding SiC/Ni reaction zone in Fig. 1; (a) SiC, (b) SiC reaction zone with alternating layers of Ni<sub>2</sub>Si+C/Ni<sub>2</sub>Si/Ni<sub>2</sub>Si+C/..., (c) metal reaction zone of Ni<sub>2</sub>Si and (d) nickel metal.



**Fig. 4.** A SEM micrograph of the SiC reaction zone after annealing at 950°C for 100 h showing a relatively wide band of Ni<sub>2</sub>Si with fine carbon precipitates.

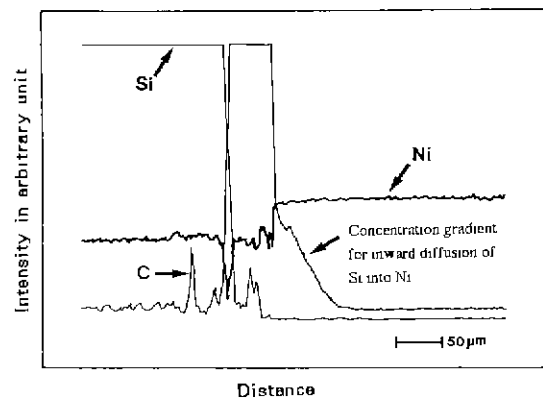
**Table 1.** Average Concentration of Silicon, Nickel and Carbon in the SiC/Ni Reaction Zone After Annealing at 950°C for 100 h.

Microstructure	wt.% Ni	wt.% Si	wt.% C
SiC	65.0	0	35.0
dark layer	64.6 ± 1.7	16.8 ± 0.3	18.6 ± 1.4
light layer	80.4 ± 0.4	20.8 ± 0.1	0

adjoining bright layer the reaction phase with the concentration of 80.4 wt% (64.9 at%) nickel, 20.8 wt% (35.1 at%) silicon is free of carbon. The concentration of carbon in all dark layers fluctuates between 20.2 and 16.3 wt%. It is noted that the value of carbon does not increase or decrease systematically. The concentration relationship of Si/Ni was confirmed to be constant and independent of the presence of carbon. This atomic ratio corresponds to Ni<sub>2</sub>Si. These alternating layers could be identified to be Ni<sub>2</sub>Si+C/Ni<sub>2</sub>Si/Ni<sub>2</sub>Si+C/.... The overall concentration of the three elements in the dark layers yields about three carbon atoms to one Ni<sub>2</sub>Si.

The microstructure adjacent to the SiC after annealing at 950°C for 100 h can be seen in a SEM micrograph in Fig. 4. The first SiC reaction zone is composed by much finer carbon precipitates in comparison to the zone further removed from the SiC reaction front. In this contact area the original microstructure of SiC can be recognized, where the precipitates are distributed texturally in the vicinity of the original SiC crystal.

On the other hand, adjacent to the unreacted nickel metal, a metal-like reaction zone is formed with large number of cracks (Fig. 2(c)) formed after the annealings in the examined temperature range between 950 and 1250°C for various times. Characteristically this zone shows the absence of carbon precipitates. As resulted from quantitative analysis with EPMA, it is proved to be Ni<sub>2</sub>Si phase. A high intensity of the EPMA line-scan (Fig. 5) at this metal reaction zone after annealing at 950°C for 100 h indicates also absence of carbon and a small intensity of silicon, which decreases continuously to un-

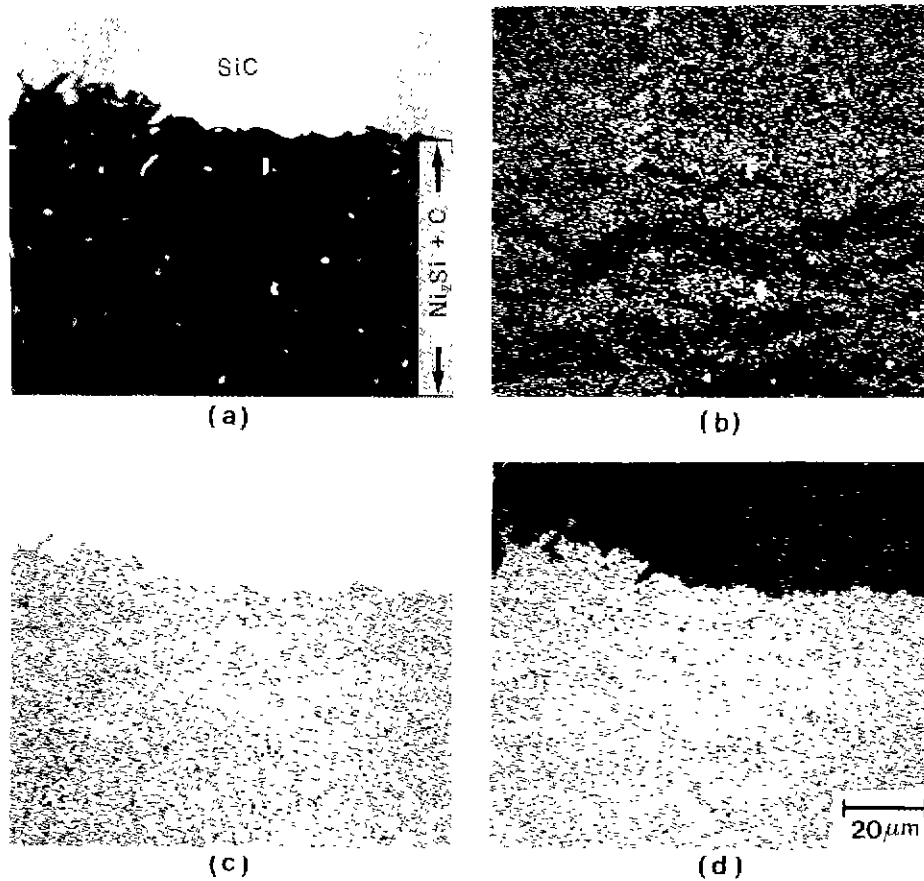


**Fig. 5.** A high intensity of the EPMA line scan for silicon, nickel and carbon at the metal reaction zone after annealing at 950°C for 100 h showing an inward diffusion zone of silicon into nickel metal with a depth of 70 μm.

reacted nickel metal. According to this profiles for silicon intensity an inward diffusion of silicon into nickel metal was detected a depth of 70 μm in Fig. 5 for this experiment.

The generation of cracks is caused by the various plastic process during cooling of the samples from the annealing temperature, due to the various thermal expansion coefficients of the formed reaction phases. It has already been suggested<sup>9,10</sup> that during cooling process of a ceramic/metal reaction couple critical tensile stress is induced at the ceramic edge and critical compressive stress at the metal edge. In the temperature ranges between 293 and 1273 K the thermal expansion coefficients are  $4.0-6.0 \times 10^{-6} \text{K}^{-1}$  for SiC,  $6.6-17.4 \times 10^{-6} \text{K}^{-1}$  for nickel and  $16.6 \times 10^{-6} \text{K}^{-1}$  for Ni<sub>2</sub>Si. The formation of the reaction products Ni<sub>2</sub>Si+C leads to a volume reduction of  $\Delta V = -0.40 \text{ cm}^3/\text{mol SiC}$ .

Details of the carbon precipitates in the alternating layers of the reaction zone after annealing at 950°C for 100 h are shown in the EPMA images of Fig. 6. The secondary electron image and X-ray maps with the distribution of the element carbon, silicon and nickel could

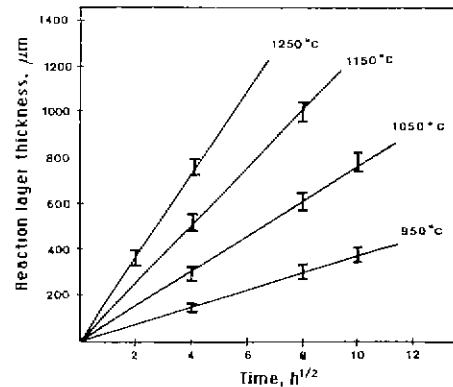


**Fig. 6.** EPMA images of the alternating layers of the SiC/Ni reaction zone after annealing at 950°C for 100 h: (a) secondary electron image, (b) carbon X-ray map, (c) silicon X-ray map and (d) nickel X-ray map.

be identified so that the carbon are revealed to be in a regular arrangement in the reaction zone only in well defined regions, where silicon and nickel are more or less uniformly distributed in all the reaction zone.

### 3. Interface chemistry

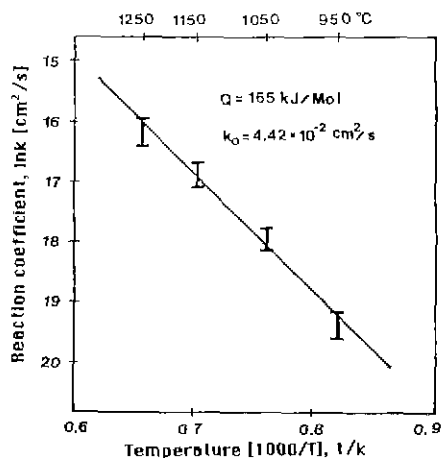
The interface chemistry was studied in the following in terms of reaction kinetics and thermodynamic, leading to the formation of nickel silicides and carbon precipitates. The kinetics of the reaction between SiC and nickel was studied for temperatures between 950 and 1250°C and times between 4 and 100 h. On the assumption that the reaction is diffusion-controlled, the thickness of the reaction zone follows a parabolic growth law. Therefore the thickness of the reaction zone are plotted in Fig. 7 for various temperatures as a function of the square root of annealing time. This linear relationship indicates that the reaction is diffusion-controlled. The relation of the logarithm of the reaction coefficients vs. the reciprocal of the absolute temperature is linear as plotted in Fig. 8. The activation energy and the frequency factor calculated from this relationship are 165 kJ/mol and  $4.42 \times 10^{-2}$  cm<sup>2</sup>/s respectively. The activation energy is similar to the values for various metal silicides formed by reactions between Si and metals at lower tem-



**Fig. 7.** Growth of the reaction thickness in SiC/Ni reaction zone vs. square root of time for various times and temperatures.

peratures.<sup>11)</sup>

Considering the Gibb's free energy ( $\Delta G$ ) for various reactions as given in the international tables<sup>12)</sup>, the reaction products between SiC and nickel could be correlated within this system. Table 2 shows Gibb's free energy of the possible reactions at 950°C. The result predicts which phases are stable at the thermodynamic equilibrium. The formation of NiSi<sub>2</sub> and Ni<sub>3</sub>C by the reaction with



**Fig. 8.** Reaction coefficients in SiC/Ni reaction zone vs. reciprocal absolute temperature [ $\ln k=f(1/T)$ ].

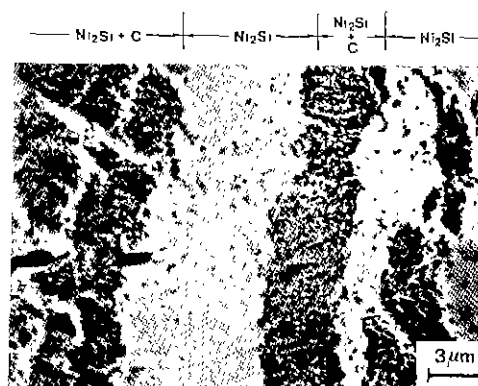
**Table 2.** Gibb's Free Energy of Possible Reactions for SiC/Ni System at 950°C.

Possible reactions	Gibb's free energy at 950°C (kJ/mol <sup>1</sup> )
$Ni + \frac{1}{2} SiC = \frac{1}{2} Ni_3C + \frac{1}{2} Si$	25.0
$Ni + 2SiC = NiSi_2 + 2C$	8.3
$Ni + SiC = NiSi + C$	-34.4
$Ni + \frac{2}{3} SiC = \frac{1}{2} Ni_3Si_2 + \frac{2}{3} C$	-44.0
$Ni + \frac{1}{2} SiC = \frac{1}{2} Ni_2Si + \frac{1}{2} C$	-46.2
$Ni + \frac{2}{3} Si = \frac{1}{2} Ni_3Si_2$	-71.3
$Ni + \frac{1}{2} SiC = \frac{1}{2} Ni_2Si$	-77.3

SiC is not thermodynamically possible at temperature of 950°C, because of the positive  $\Delta G$ -value of 25.0 and 8.3 kJ/mol respectively. At this temperature, nickel is known to react with silicon to form various silicides<sup>18-19</sup>. The  $\Delta G$ -values for the formation of Ni<sub>2</sub>Si and Ni<sub>3</sub>Si<sub>2</sub> are negative. Less negative values are calculated for the corresponding reaction  $Ni + \frac{2}{3} SiC = \frac{1}{2} Ni_3Si_2 + \frac{2}{3} C$  and  $Ni + \frac{1}{2} SiC = \frac{1}{2} Ni_2Si + \frac{1}{2} C$ , because of the energy needed for SiC decomposition. As a result, the solid state reaction between SiC and nickel could generally be described as the formation of Ni<sub>2</sub>Si+C which results in the decomposition of SiC and the precipitation of carbon.

#### IV. Discussion

The results will be discussed in the following in terms of interface structure and reaction kinetics, leading to the formation of nickel silicide and carbon precipitates in the diffusion-controlled reaction zones. The solubility and diffusion coefficients of the metals in SiC are very small, which means that the whole reaction can be described as a dissolution of SiC by nickel, yielding formation of Ni<sub>2</sub>Si+C and an extended diffusion zone with silicon and carbon enrichment of the starting nickel phase. The formation of the carbon precipitations in Ni<sub>2</sub>Si is attributed to a low



**Fig. 9.** A high magnification SEM micrograph of the SiC/Ni reaction zone after annealing at 950°C for 100 h showing the distinguishable boundary between Ni<sub>2</sub>Si and Ni<sub>2</sub>Si+C phase in the alternating layer.

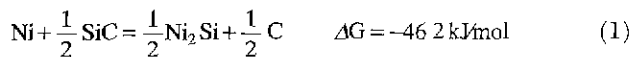
solubility of carbon in Ni<sub>2</sub>Si. In Fig. 9, a higher magnification SEM micrograph of the reaction zone of SiC/Ni after annealing at 950°C for 100 h shows an easily distinguishable boundary between the bright layer from Ni<sub>2</sub>Si and dark layer from Ni<sub>2</sub>Si+C in detail. Some carbon articles are also partially and randomly distributed in the bright area. The dark area features by the fine carbon precipitation between Ni<sub>2</sub>Si phases. The composition of the nickel silicide, Ni<sub>2</sub>Si, in the dark area is identical to that in the bright area.

As shown in Fig. 2 and Fig. 4, the width of these bands of Ni<sub>2</sub>Si and Ni<sub>2</sub>Si+C becomes smaller with increasing distance from the SiC reaction interface. The reason why the width of the band of Ni<sub>2</sub>Si and Ni<sub>2</sub>Si+C varies as a function of position from SiC reaction interface may be attributed to several competing kinetic processes, e.g., overall nucleation and growth rate of Ni<sub>2</sub>Si phases, rejecting rate of carbon from the SiC reaction front, growth rate of carbon precipitations, and diffusion rates of nickel and silicon. At the SiC reaction interface, the same amount of nickel reaching the SiC interface dissolves much more than SiC, also yielding much more than free carbon, because of the preferential formation of the reaction  $Ni + \frac{1}{2} SiC = \frac{1}{2} Ni_2Si + \frac{1}{2} C$ . Consequently, a high density of small carbon particles precipitates. Because of this high precipitation density, the effective diffusion for nickel decreases and less nickel reaches the interface, as well as less carbon forms. Since a very high density of Ni<sub>2</sub>Si+C interface is available, carbon can diffuse along these interfaces. As the reaction goes on, the Ni<sub>2</sub>Si matrix band broader and carbon supersaturation increases, until a new carbon precipitate occurs.

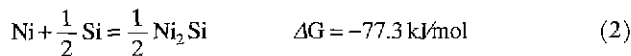
Moreover, the periodic carbon precipitation is revealed by the structure change (from fine and to aggregate structure of precipitates) according to the location of the SiC reaction interface. In the areas adjacent to the SiC interface, carbon is freshly decomposed and, therefore, exhibits primarily a fine state. For those carbon pre-

precipitates which form in areas farther from SiC reaction interface, they are decomposed earlier and therefore experience a longer heat treatment. As a result, the carbon precipitates experience a longer structural rearrangement period and tend to be more aggregate and crystallized. This driving force for the graphitization may originate from the difference of the Gibbs free energy for the possible reactions at the SiC interface and the nickel interface.

In the case of the reaction of SiC interface, SiC must be decomposed into silicon and carbon because of nickel diffusion on SiC. After decomposition of SiC, nickel reacts decomposed silicon and produces the reaction phase of Ni<sub>2</sub>Si+C. Considering the Gibbs free energy at 950°C in Table 2, at the SiC reaction interface, the possible reaction at 950°C are as follows:



On the other hand, only the reactions between silicon and nickel by the silicon diffusion into nickel and nickel diffusion into nickel silicide could be available at metal reaction interface. At this metal reaction interface, the Gibbs free energy of the possible reaction at 950°C are as follows:



Therefore, this thermodynamic driving force caused by the difference of the Gibbs free energy for the possible reactions at the SiC interface and the nickel interface causes a change of the interface structure with the carbon precipitation behaviour.

## V. Conclusions

The solid state reaction between SiC and nickel was first observed above 650°C. No carbide formation, either Ni<sub>3</sub>C or Ni<sub>2</sub>C, was detected by XRD. At 750°C, the Ni<sub>3</sub>Si<sub>2</sub> phase was transformed to be the Ni<sub>2</sub>Si phase. The thin nickel film reacted completely with SiC after annealing at 950°C for 2 h. The thermodynamically stable Ni<sub>2</sub>Si is the only observed silicide in the reaction zone up to 1050°C. At temperatures between 950 and 1050°C, the solid state reaction leads to the formation of the periodic band structure extending the typical layer sequence of SiC/Ni<sub>2</sub>Si+C/Ni<sub>2</sub>Si/Ni<sub>2</sub>Si+C/.../Ni<sub>2</sub>Si/Ni(Si)/Ni in the reaction zone. The preferential reaction of Ni+½SiC=½Ni<sub>2</sub>Si+½C is exhibited at the SiC reaction interface, and the predominant reaction of Ni+½Si=½Ni<sub>2</sub>Si is present at the nickel reaction interface.

The generation of cracks is caused by the various elastostatic processes and volume reduction of the reaction phases, SiC and metal. The reaction kinetics of growth in the thicknesses of the reaction zone shows a linear and parabolic relationship. The activation energy and

the frequency factor are proposed to be 165 kJ/mol and 4.42×10<sup>-2</sup> cm<sup>2</sup>/s. The thermodynamic driving force caused by the difference of the Gibbs free energy for the possible reactions at the SiC interface and the nickel interface causes a change of the interface structure with the carbon precipitation behaviour.

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