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Joining of Silicon Carbide Using Ni-ZrH_2 Powder Mixture †

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Abstract

Joining of pressureless-sintered SiC using (Ni + ZrH_2) powder mixture was studied using shear strength test and spectroscopic methods such as SAM and SEM-EDX. Maximum shear strength over 100 MPa was obtained in the joint using (Ni + 30mol%ZrH_2) and (Ni + 40 mol%ZrH_2) powder mixtures. Characterization of interlayer using SEM-EDX, SAM and micro-focused x-ray diffraction methods clarified that the interlayer has a cermet-like microstructure, which consists of ZrC and two nickel silicides phases.

KEY WORDS: (Silicon Carbide) (Joining) (Ni-ZrH_2 Powder)

1. Introduction

Reaction between some transition metals and SiC has been reported by some investigators[1-3]. It has been reported that some reactive metals such as titanium are effective for joining between SiCs[4]. However, details on joining mechanism remain unclear because the mechanism depends on alloying elements of additives. In the case of reaction-sintered SiC, free silicon contributes on joining of SiC but such joints can not be available for high-temperature needs because melting temperature of silicon limits the application of joint of reaction-sintered SiC. On the other hand, joint of pressureless-sintered SiC has much possibility for severe environment on account of no low melting materials. However, no good strength has been reported in the joint of pressureless SiC using high-melting insert than silicon.

In this study, joining between pressureless-sintered SiCs using Ni and ZrH_2 powders mixture as insert was investigated under a vacuum condition. Shear strengths of these joints were examined with Instrone type tensile testing machine using self-produced jig [see Fig. 1]. Characterization of these joints was performed with SEM-EDX method, micro-focused x-ray diffraction and scanning Auger microscopy (SAM) analysis.

2. Experimentals

A pressureless-sintered SiC (Kyocera Co.) was used as substrate silicon carbide. Reagent grade Ni (99.9%) and ZrH_2 (99.5%) powders were used as insert. These powders were mixed using screen oil and the mixture was painted to one-side surface of SiC rectangular plate, which was polished using diamond paste (1μm in diameter). Screen oil was evapolated in a low vacuum of 10^{-2} torr order at about 100°C. Two SiC plates, one of which was painted by (Ni + ZrH_2) powder and the other of which was not painted by the powder, were inserted into vacuum furnace so as to locate joining surface to be horizontal and (Ni + ZrH_2) powder-painted SiC plate in the under side. Temperatures in joining these SiC plates were 1773, 1823 and 1873 K, respectively. The constant load of about 0.89 kg/cm^2 was applied in the producing these SiC/SiC joints. Strength of every joint was estimated by shear strength method using tensile test machine (Autograph type IS-101, Shimadzu Co., Ltd.). Cross head speed was

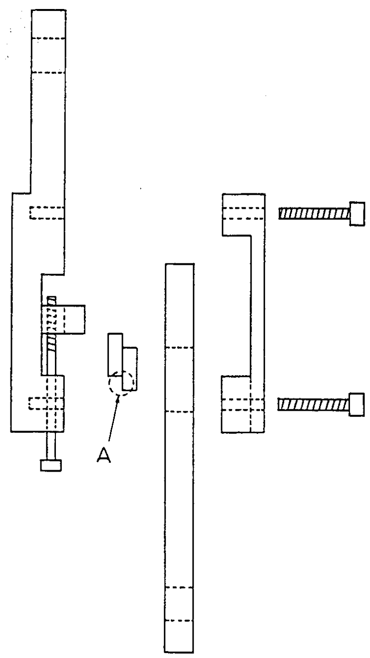

Fig. 1 Schematic picture of self-produced jig for shear strength test.

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fixed at 1 mm/min for every estimation.

Interface in SiC/(Ni + ZrH_2)/SiC joint was characterized with SEM, SAM and micro-focused XRD methods. In SEM observation, distributions of constituent elements in interlayer of joint were examined using EDX method. Phase analysis of interlayer of joint using micro-focused XRD method was performed with Cr K_α x-ray beam of 30 μm in diameter. In the SAM analysis, two dimensional distributions of some constituent elements were analyzed after Ar^+ ion sputtering for 10 to 30 min in order to remove the influence of contamination, adsorption or oxidation in specimen surface.

3. Results

Shear strength of every SiC/(Ni + ZrH_2)/SiC joint is given in fig. 2. Though the strength of joint was strongly scattered, the possibility of obtaining a shear strength in SiC/(Ni + ZrH_2)/SiC joint higher than 150 MPa is shown in this figure. In this study, higher shear strengths over 100 MPa were obtained from some joints produced using (Ni + ZrH_2) mixture with 30 mol% and 40 mol% ZrH_2. Though illustration of temperature dependence of shear strength was omitted, the strength of joint did not show so strong dependence on temperature studied and lay between 50 MPa and 150 MPa.

Characterization study on interlayer of the joint was performed using the SiC/(Ni + 30mol%ZrH_2)/SiC joint. Figure 3 shows the micro-focused x-ray diffraction pat-

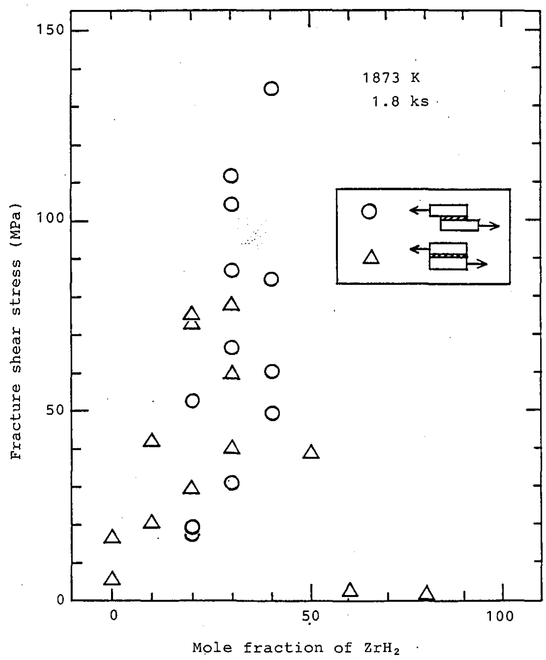


Fig. 2 Dependence of fracture shear stress upon ZrH_2 content in (Ni + ZrH_2) powder mixture.

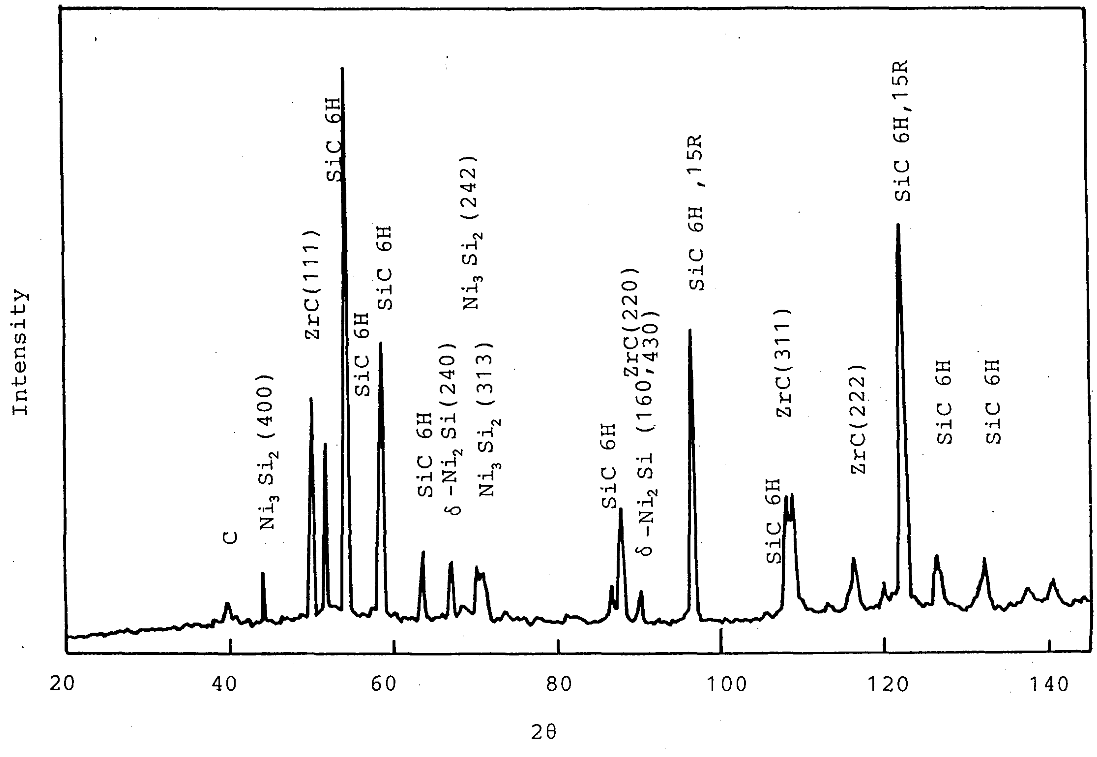
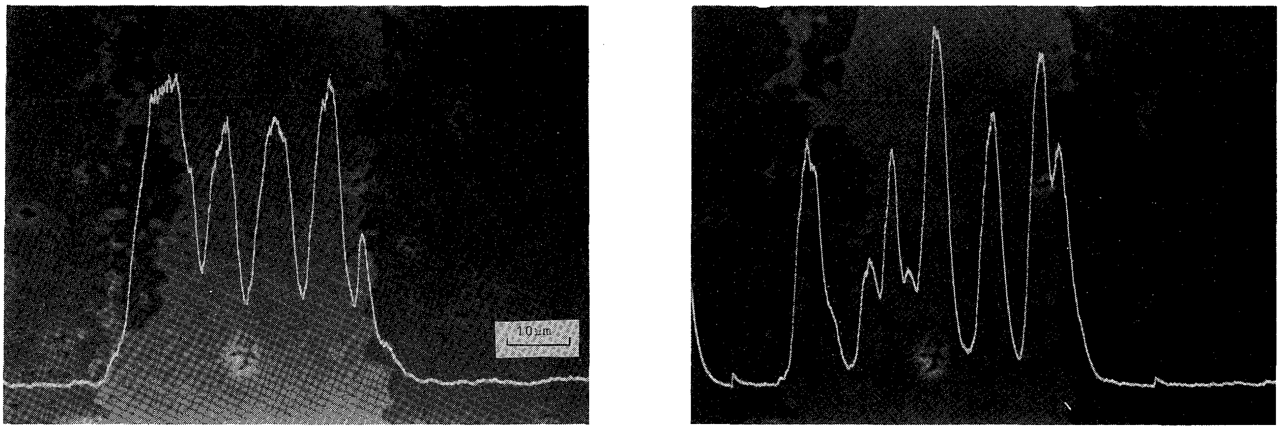


Fig. 3 X-ray diffraction pattern with micro-focusing method obtained from SiC/(Ni + 30mol%ZrH_2)/SiC joint.



Nickel Zirconium

Fig. 4 Scanning electron micrographs obtained from SiC/(Ni + 30mol%ZrH_2)/SiC joint. EDX analyses for nickel and zirconium are also shown.

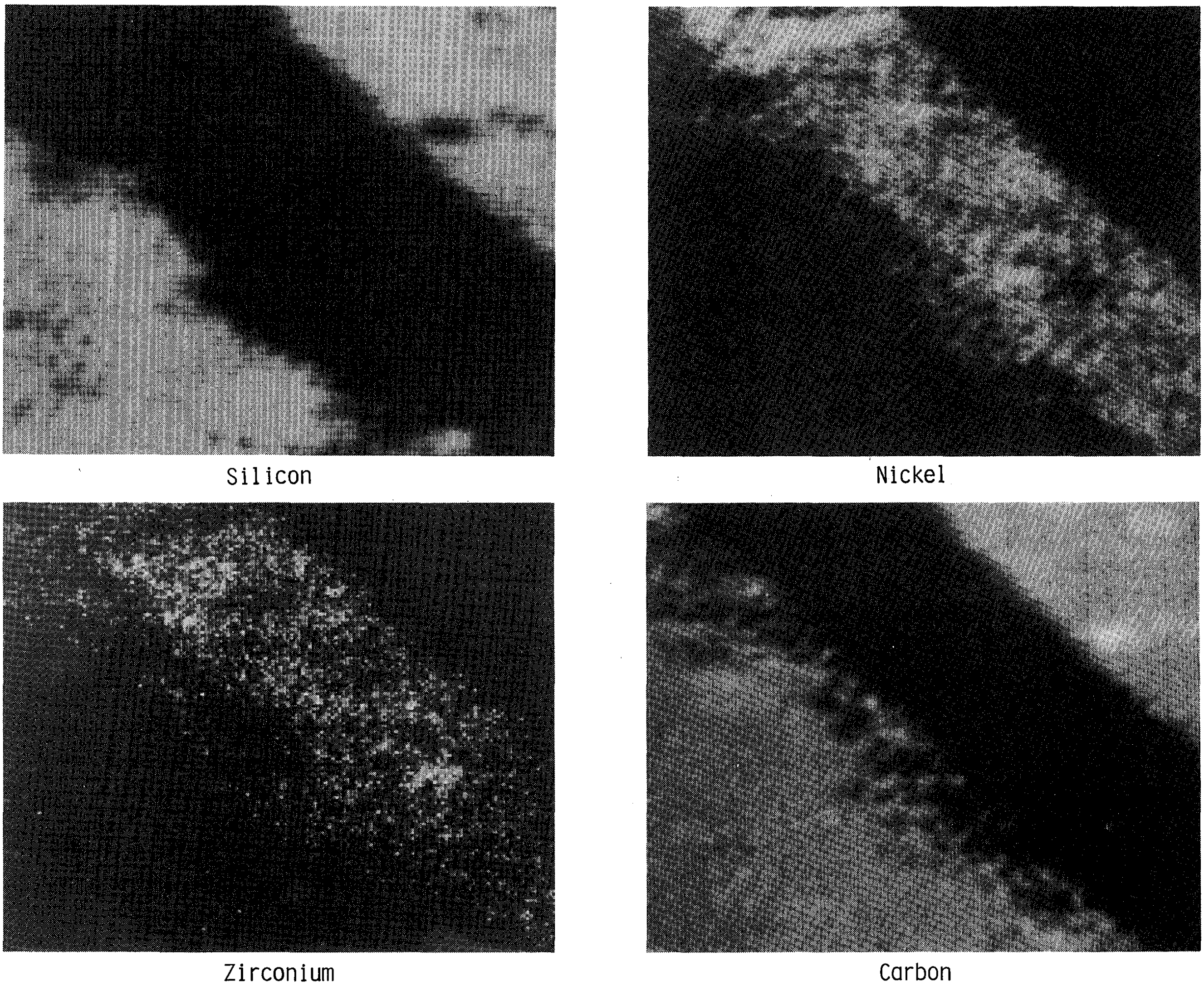


Fig. 5 Scanning auger micrographs obtained from SiC/(Ni + 30mol%ZrH_2)/SiC joint. (×500)

tern obtained from the SiC/(Ni + 30mol%ZrH_2)/SiC joint. It is detected that ZrC, δ-Ni_2Si and Ni_3Si_2 phases were formed in the interlayer of the joint. Scanning electron micrographs were given in fig. 4. From EDX analysis, it is found that interlayer shows a mottled picture and light and dark regions are zirconium- and nickel-rich, respectively.

Scanning Auger micrographs are shown in fig. 5. No good agreement between planar distributions of zirconium and carbon was obtained. Distribution of nickel did not shows so good agreement to that of silicon. It is observed that interlayer of the joint reacts to SiC substrate in some region of the interface as shown in fig. 5.

4. Discussion

Shear strength data, as shown in fig. 2, were strongly scattered, for example, from 50 MPa to 150 MPa in the SiC/(Ni + 30mol%ZrH_2)/SiC joint. One reason may be attributed to surface roughness of SiC substrate because only surfaces related to joining were polished by diamond paste. As shown in fig. 6, maximum roughness of as-received SiC substrate is about 3 μm whereas maximum roughness after polishing by diamond paste is about 0.1 μm or below. Accordingly, it is expected that difference in maximum roughness affects the sensitivity of crack initiation at the surface of SiC substrate. In the measurement of shear strength, fracture seems to be initiated very often in the non-polished surface of SiC substrate even in the case of obtaining high shear strength. This supports a considerable affect of surface roughness on shear strength.

Suitable composition of ZrH_2 in (Ni + ZrH_2) powder mixture lies between 30 to 40 mol% as shown in fig. 2. SEM observation and microfocused x-ray analyses support that interlayer is a sort of cermet-like structure which consists of ZrC and two nickel silicide phases. The structure may be responsible for high shear strength of SiC/(Ni + ZrH_2)/SiC joint and depend on the ZrH_2 composition.

From the result of micro-focused x-ray diffraction analysis, two nickel silicides were identified as δ-Ni_2Si and Ni_3Si_2 phases. The formation of δ-Ni_2Si phase shows a good agreement to the result reported by Jackson et al.[3]. Therefore, it is reasonable to interpret that carbon formed as the result of reaction between nickel and silicon carbide

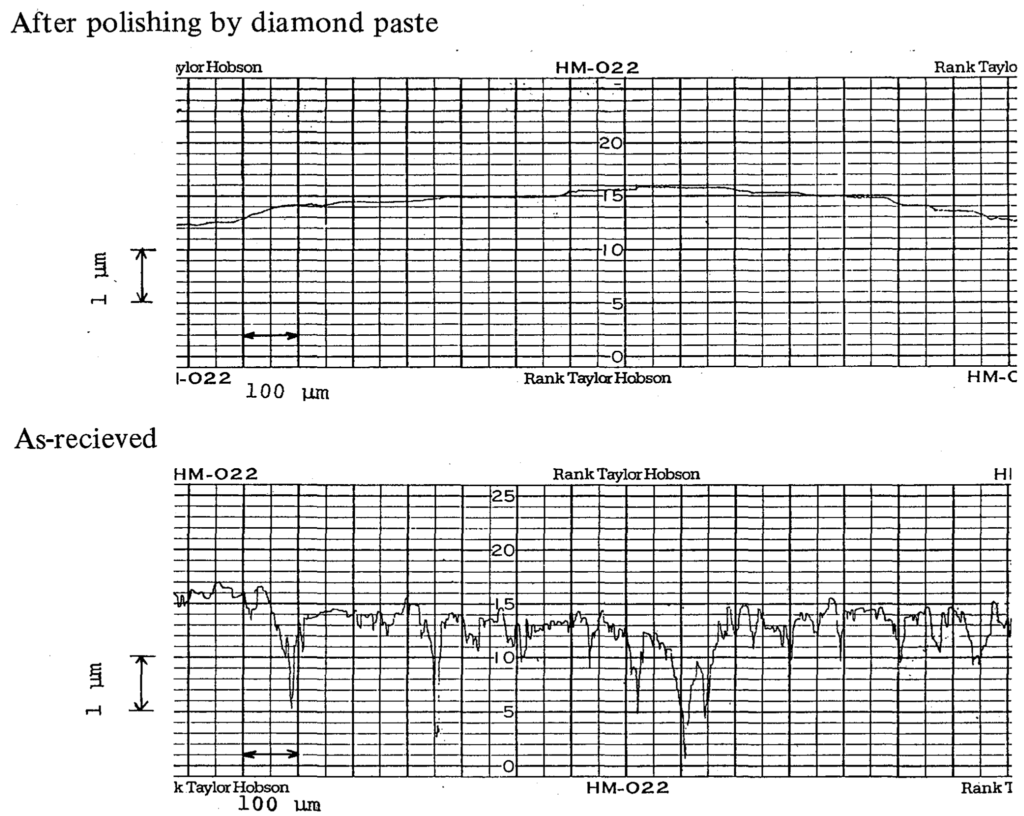


Fig. 6 Surface roughnesses of as-received SiC and SiC polished by diamond paste.

reacts with zirconium decomposed from ZrH_2. However, the details on the interfaces between ZrC and two nickel silicides remains unclear.

As shown in fig. 5, silicon and carbon are scarcely detected in SAM analysis. The results are inconsistent with the results of SEM-EDX and micro-focused x-ray analyses because carbon and silicon should be detected as the result of forming ZrC and two nickel silicide phases in the interlayer of joint. One possible reason for explaining the inconsistency is the selective sputtering. In oxide cases, oxygens can be predominantly sputtered by Ar^+ ions and metallic or suboxide phase can be formed. Some investigators indicate that the reduction of oxide by ion sputtering is related to standard free energy of formation of the oxide[5]. Selective sputtering is observed in some oxides such as TiO_2 and Fe_2O_3. For example, it has been reported in the previous paper[6] that metallic titanium or titanium monoxide is detected in plasma-sprayed titania using XPS method after Ar^+ ion sputtering. It is expected that the titania is easily reduced by Ar^+ ion sputtering because rutile phase in the titania is not well-stabilized probably as the result of rapid quenching. Selective sputtering of carbon in thin film titanium carbide on graphite has also been reported[7]. Thus, selective sputtering of carbon in ZrC phase of interlayer of $SiC/(Ni + ZrH_2)/SiC$ joint is suggested. On the other hand, possibility of selective sputtering of nickel silicide remains unclear on account of no data on silicide.

As indicated by Jackson et al.[3], graphite can be formed in the reaction between nickel and SiC. However, no detection of carbon in the interlayer of $SiC/(Ni + ZrH_2)/SiC$ joint is supported by SAM results. No detection of carbon suggests that metallic zirconium produced by the decomposition of ZrH_2 reacts with graphite completely. Further, it is suggested that residual carbon after the formation of ZrC is one of important factor for controlling the microstructure of high strength interlayer of SiC joint.

5. Summary

Joints between pressureless-sintered SiCs using ($Ni + ZrH_2$) powder mixture were produced under a vacuum condition. It is indicated that shear strength over 100 MPa was obtained in the case of insert powder mixture with 30 mol% and 40 mol% ZrH_2. Possibility of obtaining a shear strength over 150 MPa was also indicated if surface roughness of SiC substrate is controlled. From SEM-EDX, SAM and micro-focused x-ray diffraction analyses, it is clarified that interlayer of the joint is cermet-like microstructure, which consists of ZrC and two nickel silicide phases. High shear strength of the joint is produced by the cermet-like microstructure.

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