1. Introduction

Wide-band-gap (WBG) semiconductors, such as SiC and GaN, which are considered next-generation power semiconductors, have attracted significant attention among researchers.\[1–4\] In addition, WBG semiconductors have attracted attention as power modules not only because they ensure the fabrication of more compact, lightweight, and efficient power devices, but also because they enable the high-temperature operation of these devices at current density.\[5–7\] Currently, power modules fabricated using Si-IGBT can withstand high temperatures of 150°C for continuous operation and 175°C for instantaneous operation.\[8\] However, although previous studies have confirmed that devices with SiC semiconductors can operate at temperatures higher than 250°C, the bonding technique currently used in Si power modules hampers their high-temperature operation at 200°C or above. Therefore, it is important to develop a new high heat-resistant packaging technique for SiC power modules.\[9, 10\] The currently investigated packaging techniques include high-temperature lead-free solder,\[11\] transient liquid-phase (TLP) bonding,\[12\] and the sintering bonding of metal nanoparticles. Among these techniques, the sintering bonding of metal nanoparticles is considered one of the most promising techniques. When metals are converted into nanoparticles, their surface area increases, thus increasing their reactivity, which enables their bonding at relatively low temperatures. In addition, because the sintered body after bonding has the same melting point as the original metal, it can endow the bonded material with a high heat resistance.
nanoparticles and the possible increase in the resistance of Cu nanoparticles due to oxidation during the bonding process, which may affect the required bonding conditions. In addition, in both cases, voids are generated in the bonding layer during the sintering process, thus making it coarse.

In previous studies, we focused on Ni nanoparticles and their possible application for low-temperature sintering bonding. Ni has a higher melting point (1,453°C) than Ag and Cu and a lower electrical resistance than Sn-Pb eutectic solder and lead-free solder (Ni: 6.99 × 10⁻⁸ Ω·m, Sn-37Pb: 15 × 10⁻⁸ Ω·m, Sn-3Ag-0.5Cu: 11 × 10⁻⁸ Ω·m).[18, 19] Previous studies have demonstrated that Ni nanoparticles smaller than 100 nm can function as a bonding material that can be sintered below 400°C and can exhibit higher long-term resistance to temperatures higher than 250°C. [20–22]

A previous study reported that Ni nanoparticles can directly bond with Al in the atmosphere, even though Al does bond easily in soldering without flux processing.[23, 24] For example, a previous study conducted bonding experiments under different atmospheres using Si dummy chips with vacuum-deposited Al and Ni nanoparticles (Fig. 1). The results revealed that the sample bonded in atmosphere exhibited the highest shear strength on the Al surface. In addition, transmission electron microscopy (TEM) revealed that good bonding was achieved through the oxide layer with a thickness on the order of nanometers although there was an oxide layer at the bonding interface between Ni and Al. (Fig. 2) And the bonding interface exhibited uniform crystal orientation and fine bonding between elements.

However, these studies reported that cracks and voids are sometimes generated in the bonding layer of Ni nanoparticles after sintering, which is similar to the observation for other metal nanoparticles. In addition, if the thermal stress caused by the difference in the coefficient of thermal expansion (CTE) of the chip and substrate is not properly alleviated, the bonding reliability of the large chips would decrease significantly. Various studies have been conducted to solve these problems. For example, a previous study attempted to increase the bondability by adding particles of the same material and different sizes. In addition, another study aimed at suppressing the growth of micro-voids inside the bonding layer at high temperatures by adding particles with high melting points, such as SiC and tungsten.[25–28]

In this study, we proposed a stress-alleviating bonding structure that uses a composite paste composed of Ni nanoparticles and Al micro-particles with different particle sizes. Because Ni nanoparticles can directly bond with Al in the atmosphere, they acted as a bonding connection between the Al micro-particles in the bonding layer by filling the gaps created between them. In addition, because Al micro-particles are arranged in a lattice shape, the gaps in the lattice acted as a passage for the solvent gas generated during the bonding process. Furthermore, because of its excellent malleability and softness, Al may also alleviate the thermal stress on the bonding layer caused by the difference in the CTE between the chip and the substrate.

2. Experimental Methods

2.1 Materials preparation

For the experiment, a paste was prepared using Ni nanoparticles and Al micro-particles with an average size of 80 nm and 10 μm, respectively. The as-prepared paste, solvent, and binder were used as the bonding material. The weight ratio of the Ni nanoparticles and Al micro-particles in the paste was 1:1, and the volume ratio was 24:76%. This ratio indicates the dense filling of Al considering that the Al particles were uniform of diameter. A paste containing only Ni nanoparticles was also prepared and
used as a bonding material for comparison. The solid content concentration of the two types of paste was kept constant.

2.2 Preparation of samples

2.2.1 Shear test

Figure 3 shows the cross-section of the prepared sample. For the bonding strength evaluation, a TO-247 lead frame with an electro-Ni plating on the surface and three Si chips with different dimensions (2.7 × 2.7 mm, 5.0 × 5.0 mm, and 7.0 × 7.0 mm) were used. In addition, an Al electrode with a thickness of 1 µm was formed on the bonding surface of the chips by vacuum deposition. The paste was applied on the TO-247 lead frame using a metal mask made of stainless plate. Subsequently, the Si chips were placed on the applied paste, after which heat and pressure were applied to bond the sample. The thickness of the applied paste was approximately 100 µm. The bonding process was performed at a heating rate of 5°C/min, a holding temperature of 350°C, a holding time of 60 min, and a pressure of 3.5 MPa. The entire bonding process was conducted under atmospheric conditions. The number of samples for each measurement is four.

2.2.2 Observation of bonding layer

To observe the sintered bond of the bond surface, an experiment was conducted using a heat-resistant glass. Figure 4 illustrates the preparation of the sample. The paste was applied to the heat-resistant glass using a metal mask, after which a 5.0 × 5.0 mm chip was placed on the paste. Subsequently, a pressure of 0.5 MPa and a heat of 350°C were applied to the chip and maintained for 1 h. The heating rate is 5°C/min.

2.3 Sample characterization and measurement

The bonding strength was evaluated in terms of the shear strength. The bonding strength measurement was conducted at room temperature using a bond tester (4000 Plus multipurpose bond tester, Nordson Advanced Technology). The height of the tool from the surface of substrate was 100 µm, and the measurement was conducted at a traveling speed of 100 µm/sec. The fracture surface of the sample after the bonding strength test was observed using a scanning electron microscope (SEM, SU5000, Hitachi High-Technologies).

2.4 Application of the sintered bonding using Ni nano-particles/Al micro-particles composite paste to SiC device packaging

We evaluated whether the bonding technology using the Ni nanoparticles/Al microparticles is practical as a high heat resistance mounting technology. For evaluation, bonding was performed using a SiC Schottky barrier diode (SiC-SBD), and I-V characteristics were measured using a curve tracer (CS-3300, IWATSU). The I-V characteristics were measured by varying the power supply volt-

![Fig. 3 Schematic illustration of the samples bonded using (a) Ni nanoparticles paste and (b) Ni nanoparticles/Al micro-particles composite paste.](image)

![Fig. 4 Bond set for observation of the bond surface of Ni nanoparticles layer and Ni nanoparticles/Al micro-particles layer.](image)
The bonding process was carried out under air atmosphere at a heating rate of 5°C/min, a temperature of 350°C, a holding time of 60 min, and a pressure of 3.5 MPa.

2.4.1 High-temperature operation

To evaluate the bonded SiC chip, the bonding technique using the Ni/Al composite paste was applied to a SiC-SBD package and the package was subjected to high-temperature operation up to 300°C. Figure 6 shows the cross-sectional schematic illustration of the sample bonded using the Ni/Al composite paste. The Ni/Al composite paste was applied on the direct bonded copper (DBC) substrate of the bonded measurement sample. Subsequently, a SiC-SBD was placed on the paste, and heat and pressure were applied to bond the measurement sample. The SiC-SBD (1,700 V, 6.0 mm × 6.0 mm, manufactured by CREE) had an Al surface and a Ni/Ag back side.

2.4.2 High-temperature storage test

The bonding technology using the Ni nanoparticles/Al micro-particles composite paste was subjected to a long-term reliability evaluation in a high-temperature environment. To this end, the bonding technique was applied to a SiC-SBD package, and the test was conducted in an atmospheric environment at a holding temperature of 250°C for 1,000 h.

Figure 7 shows the cross-sectional illustration of the measurement sample. It was prepared by applying Ni nanoparticle/Al micro-particle composite paste on a Ni-plated TO-247 lead frame, placing a chip on it, and applying heat and pressure. The SiC-SBD chip (1,700 V, 2.1 mm × 1.6 mm, manufactured by SiCED) had an Al surface, a Ti/Ni/Ag back side.

3. Results and Discussion

3.1 Evaluation of the strength and bonding capability

Figure 8 shows a graph comparing the absolute value of the shear strength of the three chips with different sizes bonded using the Ni nanoparticles paste and the Ni/Al composite paste. For the chips bonded using the Ni nanoparticles paste, there was no increase in the shear strength with an increase in the chip size, and the strength decreased significantly at dimensions of 7.0 mm × 7.0 mm. This decrease could be attributed to the cracks and voids in the bonding layer or the thermal stress generated during the cooling process after bonding at 350°C.

In contrast, for the chips bonded using the Ni/Al composite paste, the bonding strength increased with an increase in the chip size. In addition, the shear strength at 5.0 × 5.0 mm and 7.0 × 7.0 mm was significantly higher than those of the chips bonded using the Ni nanoparticles paste. This could be attributed to the presence of fewer voids and cracks in the bonding layer and the alleviation of the stress caused by the thermal expansion difference.

Figure 9 shows a graph of the relationship between shear strength and displacement in the shear stress direction in the bonding strength test. Figure 9(a) and (b) show the bonding strength test results of the 5.0 × 5.0 mm chip.
bonded using the Ni nanoparticles paste and the Ni/Al composite paste. For comparison, the result of the bonding strength using the Ni nanoparticles paste is shown in Fig. 9(b). The (blue) curve is divided into two deformation regions: the first is the elastic deformation, and the second is the plastic deformation. The displacement of the bond containing Al micro-particles is larger than that of the bond formed using the Ni nanoparticles paste. This larger displacement of the Ni/Al composite paste bond could be attributed to the shear stress applied to the Al micro-particles, which caused them to stretch and suffer plastic deformation. These results indicate that the presence of Al micro-particles in the bonding layer significantly increased the bonding strength and strain deformation of the bonding layer.

### 3.2 Cross-sectional observation of the bonding layer

Figure 10 shows the SEM cross-sectional images of the samples bonded using the Ni nanoparticles paste and Ni/Al composite paste. The insets show the corresponding higher magnification SEM images of samples. Large voids and cracks can be observed in the cross-sectional images of the samples bonded using Ni nanoparticles; however, no cracks or voids can be observed in the images of the sample bonded using Ni/Al composite paste. In addition,
the SEM cross-sectional images reveal that the Al micro-particles are almost evenly distributed in the sintered bonding layer. Generally, if the uniform Al particles are distributed in the bond layer, a filling rate of 74% corresponds to the close-packed structure. In this study, as the volume ratio of the Al particles was approximately 76%, the filling rate was set at 76% to form a nearly close-packed lattice.

### 3.3 Surface observation after shear test

Figure 11 shows the optical microscope images of the fractured surfaces of the samples after the shear test. Figure 11(a) and (b) show the fractured surface of the samples bonded using the Ni nanoparticles paste, whereas (c) and (d) show the fractured surface of the samples bonded using the Ni/Al composite paste. Figure 11(a) and (c) show the fractured surface of the chip side of the samples, and Fig. 11(b) and (d) show the fractured surface of the substrate side of the sample. As shown in the image, fractures can be observed on the Ni surface of the TO-247 lead frame side and close to the bonding layer, whereas no fracture was observed on the Al surface of the chip side. This suggests that the Ni surface exhibited a better bondability.

![Fracture surfaces after shear test of bonded 5.0 mm x 5.0 mm chips.](image)

- (a) Si chip-Ni bonding layer
- (b) substrate-Ni bonding layer
- (c) Si chip-Ni/Al bonding layer
- (d) substrate-Ni/Al bonding layer

**Fig. 11** Fracture surfaces after shear test of bonded 5.0 mm x 5.0 mm chips. (a) Si chip-Ni bonding layer, (b) substrate-Ni bonding layer, (c) Si chip-Ni/Al bonding layer, and (d) substrate-Ni/Al bonding layer.
than the Al surface.

Figure 12 shows the SEM images and the result of the EDX elemental analysis of the fracture surface. Very few spherical Al particles can be observed on the fracture surface after the shear test, as most of them were stretched into plates (Fig. 12(a)). In addition, the elemental analysis mapping (Fig. 12(b), (c)) detected Ni elements between the Al layer. These results suggest that when shear stress was applied to the Al particles, the particles stretched, plastically deformed, and finally, fractured.

### 3.4 Comparison of the bonding layer of Ni nanoparticles with and without Al micro-particles bonding

The observation results of the pilled surface are shown in Fig. 13. Figure 13(a) and (b) show the surface of the samples prepared using only the Ni nanoparticles paste and Ni/Al composite paste, respectively. Large voids and cracks can be observed close to the center of the surface.
of the sample bonded using the Ni nanoparticles paste. In contrast, the bonding layer of the sample bonded using the Ni/Al composite paste was a uniform without large voids or cracks.

As schematically shown in Fig. 14, these results indicate that the solvent that was evaporated during the bonding process using the Ni nanoparticles paste was trapped inside the bonding layer, thus generating voids and cracks.

In contrast, the distributed structure formed by the Al micro-particles secured a path for the escape of the evaporated solvent, thus reducing the generation of large voids and cracks.

3.5 Characterization of the bonded SiC-SBD in high temperature environment

Figure 15 (a) shows the I-V measurement result of the SiC-SBD chip before and after the bond. The inset in the
Fig. 15 (a) is an enlarged view of the voltage characteristics when the current starts to flow. The image indicates that the curves have a similar tendency and are mutually close. As a result, there were no changes in the I-V characteristics of the SiC-SBD chip before and after the bond.

Figure 15 (b) shows the result of the I-V curve with each heating temperature. As shown in the graph, the forward voltage shifts to the left with an increase in temperature, which indicates that the diode is temperature-dependent and operates normally even at a heating temperature of 300°C.

3.6 Evaluation of the bonded SiC-SBD after the high-temperature storage test

Figure 16 shows the I-V characteristics of the bonded SiC-SBD after the high-temperature storage for different time duration. The result revealed that the threshold voltage after the bonding was approximately 0.8 V, and there was no significant difference in the voltage at each holding time of the high-temperature storage test. In addition, the I-V curves before and after the high-temperature storage test in the graph Fig. 16 are close, and have similar tendencies.

Figure 17 shows the graph of the operating resistance, which was determined from the I-V characteristics measured during the high-temperature storage test of three samples. The results revealed that there was only a slight change in the resistance of the samples at different holding times of the high-temperature storage test at 250°C. Ultimately, the resistance increased by 4.3% after 1,000 h, with no significant increase in the resistance after that period. This result suggests that the high-temperature storage test had no significant effect on the interior of the bonding layer, indicating the stability of the bonding reliability.

4. Conclusions

In this study, we investigated the bondability of a composite paste prepared using Ni nanoparticles and Al micro-particles and obtained the following results. The properties of the fine bonding layer formed by this paste indicates its promising potential for application in the packaging of SiC power devices as a bonding structure with a stress alleviation effect. The major results of the study are as follows:

1. The addition of sub-micron sized Al particles into the Ni nanoparticles paste can reduce the generation of voids and cracks in the bonding layer.
2. The bonding strength of the sample bonded using the Ni/Al composite paste was similar to that bonded using Ni nanoparticles paste, indicating that the Ni nanoparticles were finely bonded with the Al particles after sintering.
3. The stress-strain curve indicates that the Al particles distributed in the bonding layer was susceptible to plastic deformation, and the fracture surface observation after the shear test also confirmed that the Al particles stretched and suffered plastic deformation.
4. A SiC-SBD package was subjected to a high-temperature storage test, and it operated normally even at 300°C.
5. There was no significant increase in the operating resistance of the sample subjected to high-temperature storage test at 250°C, indicating that the bond...
using Ni/Al composite paste had no significant effect on the electrical properties of the sample. This result suggests that this paste is highly reliable even when exposed to high-temperature environments for a long period.

Based on these results, we plan to conduct thermal cycling tests in the future to evaluate the joint reliability for practical use.

References
[1] T. P. Chow and R. Tyagi, “Wide bandgap compound semiconductors for superior high-voltage unipolar power devices,” IEEE Trans. Electron Devices, Vol. 41, pp. 1481–1483, 1994.
[2] J. L. Hudgins, G. S. Simin, E. Santi, and M. A. Khan, “An assessment of wide bandgap semiconductors for power devices,” IEEE Trans. Power Electron, Vol. 18, pp. 907–914, 2003.
[3] J. Millán, P. Godignon, X. Perpiñà, A. P. Tomás, and J. Rebollo, “A Survey of Wide Bandgap Power Semiconductor Devices,” IEEE Trans. Power Electron, Vol. 29, pp. 2155–2163, 2014.
[4] X. She, A. Q. Huang, Ó. Lucia, and B. Ozpineci, “Review of Silicon Carbide Power Devices and Their Applications,” IEEE Trans. Ind. Electron, Vol. 64, pp. 8193–8205, 2017.
[5] T. Funaki, J. C. Balda, J. Junghans, A. S. Kashyap, H. A. Mantooth, F. Barlow, T. Kimoto, and T. Hikihara, “Power conversion with SiC devices at extremely high ambient temperatures,” IEEE Trans. Power Electron, Vol. 22, pp. 1321–1329, 2007.
[6] P. Ning, F. Wang, and K. D. T. Ngo, “High-temperature SiC power module electrical evaluation procedure,” IEEE Trans. Power Electron, Vol. 26, pp. 3079–3083, 2011.
[7] S. Ji, Z. Zhang, and F. Wang, “Overview of High Voltage SiC Power Semiconductor Devices: Development and Application,” CES Transactions on Electrical Machines and Systems, Vol. 1, pp. 254–264, 2017.
[8] K. Sheng, L. C. Yu, J. Zhang, and J. H. Zhao, “High temperature characterization of SiC BJTs for power switching applications,” Solid State Electron, Vol. 50, pp. 1073–1079, 2006.
[9] V. R. Manikam and K. Y. Cheong, “Die Attach Materials for High Temperature Applications: A Review,” IEEE Trans. Compon., Packag., Manuf. Technol., Vol. 1, pp. 457–478, 2011.
[10] L. Coppola, D. Huff, F. Wang, R. Burgos, and D. Boroyevich, “Survey on High Temperature Packaging Materials for SiC-Based Power Electronics,” Proc. PESC, Orlando, FL, pp. 2234–2240, 2007.
[11] G. Zeng, S. McDonald, and K. Nogita, “Development of high-temperature solders: Review,” Microelectron. Reliab, Vol. 52, pp. 1306–1322, 2012.
[12] H. A. Mustain, W. D. Brown, and S. S. Ang, “Transient Liquid Phase Die Attach for High Temperature Silicon Carbide Power Devices,” IEEE Trans. Compon. Packag. Technol., Vol. 33, pp. 563–570, 2010.
[13] T. G. Lei, J. N. Calata, G. Q. Lu, C. Xu, and S. Luo, “Low-Temperature Sintering of Nanoscale Silver Paste for Attaching Large-Area (>100 mm²) Chips,” IEEE Trans. Compon. Packag. Technol., Vol. 33, pp. 98–104, 2010.
[14] A. Hu, J. Y. Guo, H. Alarifi, G. Patane, Y. Zhou, G. Compagnini, and C. X. Xu, “Low temperature sintering of Ag nanoparticles for flexible electronics packaging,” Appl. Phys. Lett, Vol. 97, 153117, 2010.
[15] P. Peng, A. Hu, B. Zhao, A. P. Gerlich, and Y. N. Zhou, “Reinforcement of Ag nanoparticle paste with nanowires for low temperature pressureless bonding,” J. Mater. Sci, Vol. 47, pp. 6801–6811, 2012.
[16] Y. Li, D. Li, C. Li, H. Wang, D. Shen, L. Liu, and G. Zhou, “Annealing-induced highly-conductive and stable Cu-organic composite nanoparticles with hierarchical structures,” J. Alloys Compd, Vol. 636, pp. 1–7, 2015.
[17] J. Li, X. Yu, T. Shi, C. Cheng, J. Fan, S. Cheng, G. Liao, and Z. Tang, “Low-Temperature and Low-Pressure Cu-Cu Bonding by Highly Sinterable Cu Nanoparticle Paste,” Nanoscale Res Lett, pp. 1–6, 2017.
[18] T. Farrell and D. Greig, “The electrical resistivity of nickel and its alloys,” J. Phys. C: Solid State Phys, Vol. 1, p. 1359, 1968.
[19] K. Ikeda, “Electrical Resistivity of Nickel, Cobalt and Their Alloys,” Trans. Jpn. Inst. Metals, Vol. 29, pp. 185–190, 1988.
[20] N. Kato, S. Hashimoto, T. Iizuka, and K. Tatsumi, “High-Temperature-Resistant Interconnections Formed by Using Nickel Micro-plating and Ni Nanoparticles for Power Devices,” Trans. Jpn. Inst. Electron. Packaging, Vol. 6, pp. 87–92, 2013.
[21] Y. Tanaka, S. Hashimoto, T. Iizuka, K. Tatsumi, N. Matsubara, S. Ishikawa, and M. Tanaka, “High-temperature-resistant interconnection using nickel nanoparticles,” International Microelectronics
Assembly and Packaging (IMAPS), pp. 561–565, 2014.

[22] Y. Tanaka, K. Ota, H. Niyano, Y. Shigenaga, T. Iizuka, and K. Tatsumi, “High Temperature Resistant Packaging for SiC Power Devices Using Interconnections Formed by Ni Micro-Electro-Plating and Ni Nanoparticles,” Proceedings of 65th IEEE Electronic Components and Technology Conference (ECTC), pp. 1371–1376, 2015.

[23] K. Tatsumi, Y. Tanaka, T. Iizuka, K. Wada, M. Fukumori, I. Morisako, Y. Jeongbin, and N. Murakawa, “High temperature resistant interconnection for SiC power devices using Ni micro-electro-plating and Ni nano particles,” Proceedings of 7th Electronic System-Integration Technology Conference (ESTC), 2018.

[24] T. Iizuka, Y. Tanaka, K. Kamei, M. Inagaki, N. Murakawa, and K. Tatsumi, “High-temperature-resistant interconnection by using Nickel Nano-particles for power devices packaging,” Proceedings of 24th International Symposium on Semiconductor Manufacturing, 2016.

[25] K. Tatsumi, “METAL JOINING STRUCTURE USING METAL NANOPARTICLES AND METAL JOINING METHOD AND METAL JOINING MATERIAL,” U.S.9, 960, 140 B2. May 1, 2018.

[26] K. Suganuma, S. Sakamoto, N. Kagami, D. Wakuda, K.-S. Kim, and M. Nogi, “Low-temperature low-pressure die attach with hybrid silver particle paste,” Microelectronics Reliability, Vol. 52, pp. 375–380, 2012.

[27] H. Zhang, S. Nagao, and K. Suganuma, “Addiction of SiC Particles to Ag Die-Attach Paste to Improve High-Temperature Stability; Grain Growth Kinetics of Sintered Porous Ag,” Journal of ELECTRONIC MATERIALS, Vol. 44, pp. 3896–3903, 2015.

[28] K. Sugiura, T. Iwashige, J. Kawai, K. Tsuruta, C. Chen, S. Nagano, H. Zhang, T. Sugahara, and K. Suganuma, “Prominent Interface Structure and Bonding Material of Power Module for High Temperature Operation,” Proceeding of The 29th International Symposium on Power Semiconductor Devices and ICs, pp. 491–494, 2017.

[29] T. C. Hales, “A proof of the Kepler conjecture,” Annals of Mathematics, Vol. 162, pp. 1065–1185, 2005.

---

Yasunori Tanaka
Tatsumasa Wakata
Norihiro Murakawa
Tomonori Iizuka
Md. Mijanur Rahman
Kohei Tatsumi