Abridged version

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Development of AlN Substrate with High Thermal Conductivity

Makoto Yokoi, Takao Kanemaru, Masato Kumagai, Tadashi Nakano, Toshihiko Funahashi, Yutaka Yoshii

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Aluminum nitride (AlN), an attractive candidate for the high performance substrate of the next generation, has been developed, and metallization techniques have also been advanced. High thermal conductivity AlN substrate with more than 180 W/m･K has successfully been obtained by using high purity, especially low oxygen content AlN powder, and by decreasing impurities, attaining grain growth and eliminating pores completely in the sintered body. Development of an excellent organic binder, tape casting, binder burn-out and the sintering process has resulted in the highly efficient manufacturer of the AlN substrate. AlN is a suitable substrate for mounting highly integrated, high power and large-sized semiconductor devices. With the above mentioned development of the manufacturing process and metallization techniques, large-scale practical applications of the AlN substrate have been expected.

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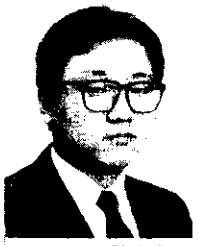
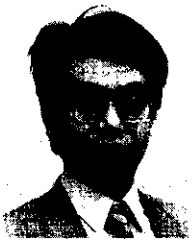
Development of AlN Substrate with High Thermal Conductivity*


Makoto Yokoi
Senior Researcher,
New Materials
Research Center,
High-Technology
Res. Labs.

Takao Kanemaru
New Materials
Research Center,
High-Technology
Res. Labs.

Masato Kumagai
Dr. Engi., Senior
Researcher,
New Materials
Research Center,
High-Technology
Res. Labs.


Tadashi Nakano
Senior Researcher,
New Materials
Research Center,
High-Technology
Res. Labs.

Toshihiko Funahashi
Senior Researcher,
New Materials
Research Center,
High-Technology
Res. Labs.

Yutaka Yoshii
Senior Researcher,
New Materials
Research Center,
High-Technology
Res. Labs.

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Aluminum nitride (AlN), an attractive candidate for the high performance substrate of the next generation, has been developed, and metallization techniques have also been advanced. High thermal conductivity AlN substrate with more than 180 W/m·K has successfully been obtained by using high purity, especially low oxygen content AlN powder, and by decreasing impurities, attaining grain growth and eliminating pores completely in the sintered body. Development of an excellent organic binder, tape casting, binder burn-out and the sintering process has resulted in the highly efficient manufacture of the AlN substrate. AlN is a suitable substrate for mounting highly integrated, high power and large-sized semiconductor devices. With the above mentioned development of the manufacturing process and metallization techniques, large-scale practical applications of the AlN substrate have been expected.

1 Introduction

Electric equipment is getting increasingly sophisticated in specification toward higher speed, smaller size, lighter weight, and higher reliability. Contrarily, semiconductor element devices are rapidly changing toward larger size, higher integration, and higher output, while wiring circuit is toward small size, shorter length, and lower resistivity. This requires substrate to have increasingly higher grade and diversified properties as base plate of semiconductor element devices.

In particular, an increase in thermal volume resulting from high integration and high output of element devices poses serious problems, which would lead to mulfunctioning of element devices and marked reduction in their reliability. Heat radiation can be performed by (1) cooling fans, (2) forced air cooling, or water cooling mechanism, (3) use of high thermal conductivity substrates. The use of high thermal conductivity substrates is especially in a growing demand as it permits miniatuarization and light weight.

Meanwhile, the growing sizes of semiconductor devices have led to serious problems of mismatch between such devices (especially those made of Si) and substrates in terms of thermal expansion and formation of cracks, thus impairing the reliability of semiconductor devices. This calls for a need for substrates having a thermal expansion coefficient as close to that of Si ($3.5 \times 10^{-6}/°C$) as possible. To meet this need, various matrials including ceramics are being developed.[1)]

This report describes an AlN substrate which has been developed at Kawasaki Steel, with emphases on sintering mechanism clarified, and findings on microstructure control for achieving high thermal conductivity, and metallizing characteristics.

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2 Technical Trend in Ceramic Substrates

2.1 Present State and Problems

Materials for IC substrates are broadly classified into plastics, ceramics, and metallic composite materials. To be effective substrates these materials must have the following properties: (1) high electrical resistivity, (2) high breakdown voltage, (3) good dielectric properties, (4) high mechanical strength, (5) good formability (dimensional accuracy), (6) heat resistance, (7) high thermal conductivity, (8) a thermal expansion coefficient compatible with surrounding materials, (9) high chemical stability, and (10) low cost.[2] In consideration of the above properties, materials for substrates are chosen according to their required application.

Among ceramics, alumina accounts for more than 90% of ceramic substrates since it satisfies almost all of the above requirements. However, since the trend in electronic devices is towards higher speeds, miniaturization and higher reliability, there have been many applications for which the conventional alumina substrates are not suitable. For this reason, the development of next-generation substrates is being concentrated in two major directions; i.e., in substrates having a high thermal conductivity and in multilayer ceramic substrates with built-in capacitors and resistors.

2.2 Substrates with High Thermal Conductivity

Ceramics with high electrical insulation conduct heat mainly by means of phonons. The thermal conductivity of a single crystal of Adamantin structure is given by the following formula[3]:

$$\kappa = BM\delta\theta_D^3/T\gamma^2 \quad \cdots\cdots (1)$$

where M: Average molecular weight of unit cell
δ: Cubic root of occupied volume per atom in unit cell
θ_D: Debye temperature
T: Absolute temperature
γ: Grüneisen constant
B: Constant

Substances with high $M\delta\theta_D^3$, i.e. substances having light atomic weight, strong atomic bonds, and a simple crystal structure, show great promise for possessing high thermal conductivity.

Table 1 lists the properties of high thermal conductivity ceramic substrates which are in use or have been developed, and compares them with those of alumina substrates. Diamond and c-BN have especially high thermal conductivity and also have other superior substrate properties. Because of high prices, they are, however, used in very limited fields, such as for heat sinks of laser diodes. Although SiC is superior to alumina in thermal conductivity and has a higher thermal expansion coefficient, it also has a low breakdown voltage and high dielectric constants. In addition, SiC substrates can be produced only by the hot pressing method, therefore, it is difficult to reduce manufacturing costs. BeO, which has high thermal conductivity and is equal or superior to alumina in electrical, mechanical and chemical properties, has long been used in substrates for high-frequency devices. Because of its strong toxicity, BeO is known to have limits to wide applications.

AlN is superior to alumina in thermal conductivity and has a similar thermal expansion coefficient to Si, and also its other electrical and mechanical properties such as breakdown voltage and bending strength are equal or superior to alumina. It might be said that AlN is a well-balanced material. AlN permits continuous sheet forming and pressureless sintering and is equal to alumina in terms of mass production. The problem, however, is that only about a half of 320 W/m·K (the theoretical value of thermal conductivity) is obtained from commercial AlN substrates, due to the scattering of phonons, which is ascribed to various causes[4] as

Table 1 Typical properties of high thermal conductivity ceramic substrates

		Al_2O_3	AlN	SiC	BeO	c-BN
Thermal conductivity (RT)	(W/m·K)	20	100~180	270	250	1 300
Electrical resistivity (RT)	(Ω·cm)	$>10^{14}$	$>10^{14}$	$>10^{18}$	$>10^{14}$	$>10^{14}$
Insulation voltage (RT)	(kV/cm)	100	140~170	0.7	100	100~140
Dielectric constant (RT·1 MHz)	ε	8.5	8.8	42	8.5	6.7
Dielectric loss (1 MHz)	(10^{-4} tan δ)	3	5	500	3	4~7
Thermal expansion coeff.	(10^{-6}/K)					
RT—200°C			3.5			
RT—400°C		7.3	4.5	3.7	7.3	7.2
Density	(g/cm³)	3.9	3.3	3.2	3.9	2.9
Young's modulus	(10^4 kg/mm²)	3.7	2.8	4.0	3.7	3.2
Modulus of rupture	(kg/mm²)	20~30	30~50	45	20~30	17~23
Sintering pressure		normal	normal	(hot pressing)	normal	ultra high
Additives		SiO_2, MgO	CaO, Y_2O_3	BeO		

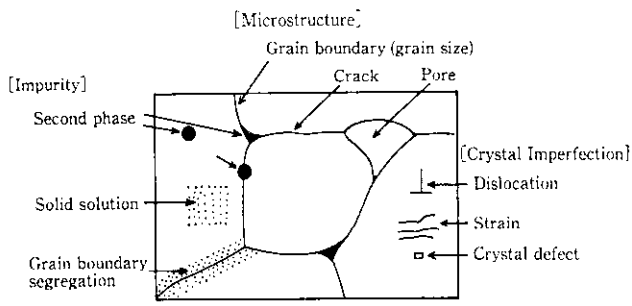

Fig. 1 Factors for decreasing thermal conductivity of ceramics (by H. Yanagida and S. Hishida[6])

shown in **Fig. 1**.

To mass produce high thermal conductivity AlN with high reproducibility, it is necessary to remove the causes of phonon scattering, particularly, to reduce the amount of solid-solute oxygen included in the grain, precipitates, pores, and grain-boundary gaps.

3 Manufacturing Process for AlN Substrates

AlN substrates can be produced by the pressureless sintering of a sheet obtained by the doctor-blade method, which is the same continuous tape casting method used for alumina substrates. Under the doctor-blade method[5,6], a slurry, composed of ceramic powder, dispersant, binder, plasticizer and organic solvent, is fed onto a moving film, the film is continuously formed using a knife-like edge called a doctor-blade, and, after drying, the formed film is reeled to produce thin sheets 1 m in width and less than 1 mm in thickness. However, the same techniques as for alumina substrates cannot be applied to some types of organic binder, and the methods for their burn-out and firing, therefore, techniques that permit mass production were established by optimizing these items.[7]

4 Densification Process of AlN

AlN is difficult to sinter. In order to sinter this material under normal pressure, it is generally necessary to add sintering aids such as alkaline earth metal compounds or rare earth metal compounds.[8,9] The sintering mechanism of AlN containing these aids is liquid-phase sintering. In other words, all of these additives react with Al–O compounds, present on the surface of AlN particles, in the heating process to form liquid phases. The existence of liquid phases accelerates mass transfer, particle rearrangement, etc., thus facilitating the progress of sintering. Although the sintering temperature, holding time, heating rate and atmospheric pressure are also factors influencing densification, special attention was paid here to the atmospheric conditions during firing which are more important than the other factors. The effect of the sintering atmosphere on the sintering process of AlN is described below by reference to a case where Y_2O_3 was added as the sintering aid.

4.1 Experimental Procedure

Y_2O_3 was added to AlN powder (mean diameter: 0.8 μm) and, after tape casting and binder burn-out, the sheet was sintered at 1 550 to 1 900°C for 0 to 4 h in a nitrogen flow under normal pressure.

Firing was conducted in both a reducing atmosphere and a nonreducing atmosphere to enable a comparison to be made between the two. Since a graphite furnace was used, the atmosphere in the furnace was a reducing atmosphere. Nonreducing atmosphere was prepared by shutting off the burnout body from the reducing atmosphere by improving the material for the vessel.

4.2 Results and Discussion

Figure 2 shows changes in density at various temperatures in a reducing atmosphere and a nonreducing atmosphere. The densification rate was low in the reducing atmosphere and complete densification was not achieved. In the nonreducing atmosphere, a rapid densification occurred at about 1 800°C and a dense sintered body was obtained at 1 900°C. Changes in the oxygen and yttrium contents in the densification process are shown in **Fig. 3**. The yttrium content did not change in either of the two atmospheres. This shows that the vaporization of yttrium did not occur. In the reducing atmosphere, the oxygen content began to decrease remarkably at 1 550°C and was present only as the oxygen portion of Y_2O_3 at 1 800°C and above. **Figures 4** and **5** show changes in the grain boundary phase when firing was conducted in the two atmospheres. In the nonreducing atmosphere, conjugated oxides such as $Al_2Y_4O_9$ and $AlYO_3$, which are liquid-phase components, were present. In the reducing

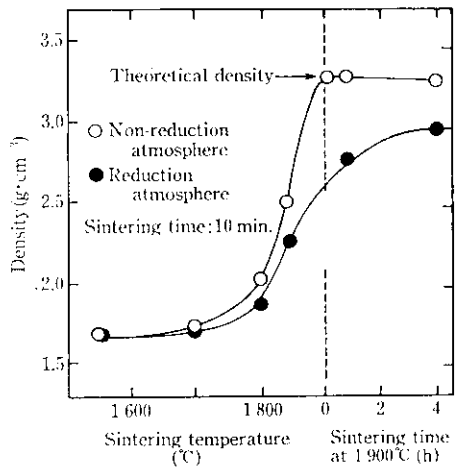

Fig. 2 Densities of AlN ceramics as a function of sintering temperature and time under different atmosphere

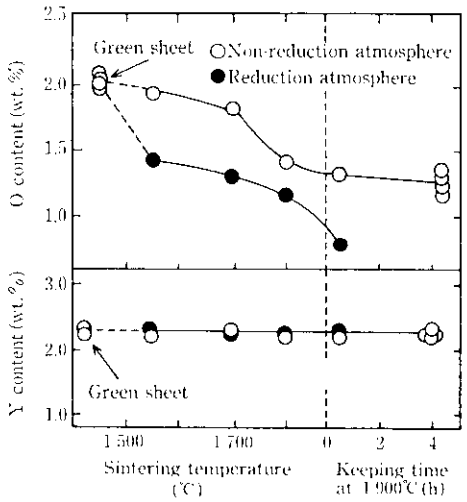

Fig. 3 Change in oxygen and yttrium content as a function of sintering temperature and keeping time

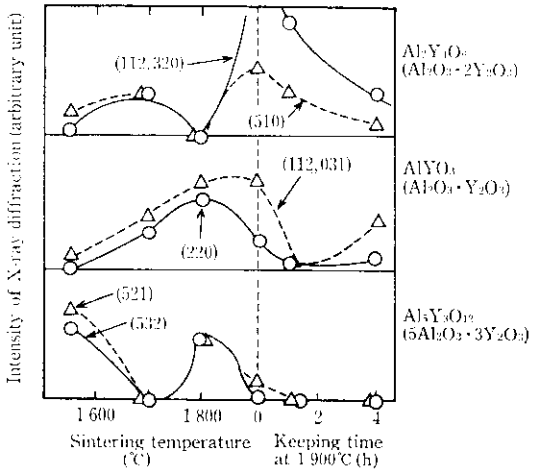

Fig. 5 Change in grain boundary phase of AlN ceramics as a function of sintering temperature under non-reduction atmosphere (sintering time: 10 min)

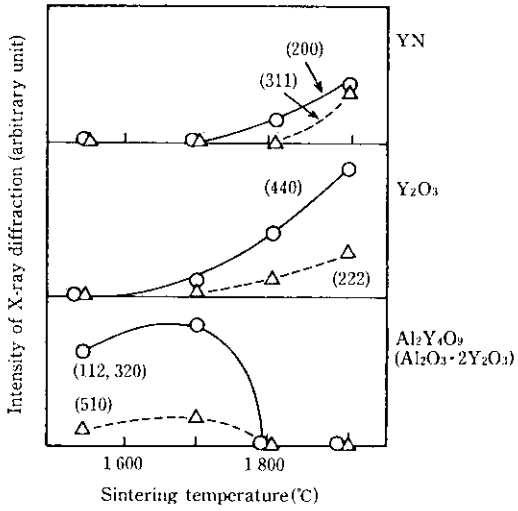

Fig. 4 Change in grain boundary phase of AlN ceramics as a function of sintering temperature under reduction atmosphere (sintering time: 10 min)

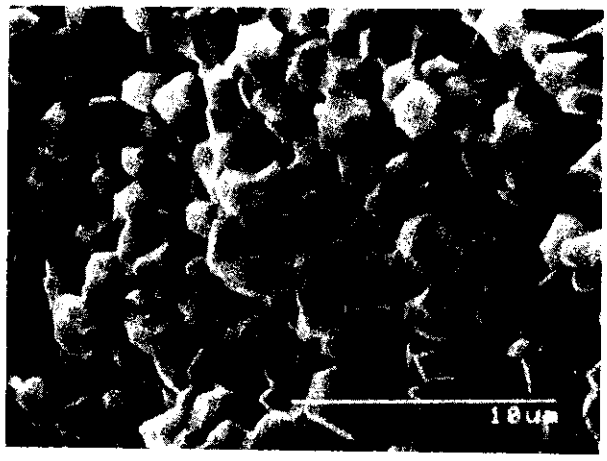

Photo 1 SEM micrograph of AlN after sintering under non-reduction atmosphere

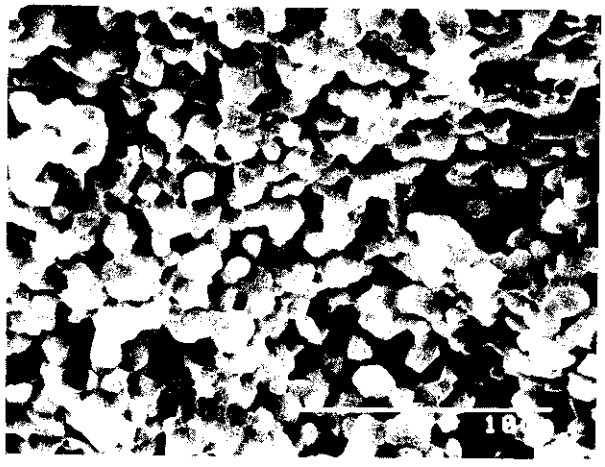

Photo 2 SEM micrograph of AlN after sintering under reduction atmosphere

atmosphere, however, the liquid-phase components disappeared almost completely and YN and high-melting-point Y_2O_3 appeared. **Photos 1** and **2** show the SEM micrographs of samples fired at 1 850°C. Grain growth proceeded well and polygonal grains of AlN were formed in the nonreducing atmosphere. The grains were small and round and many pores were observed in the reducing atmosphere. Thus there is a great difference noticed in the condition of progress of sintering.

The chemical composition of the liquid-phase at the grain boundaries in the sintering process was estimated according to the following method. On the assumption that the oxygen determined from the analytical value of

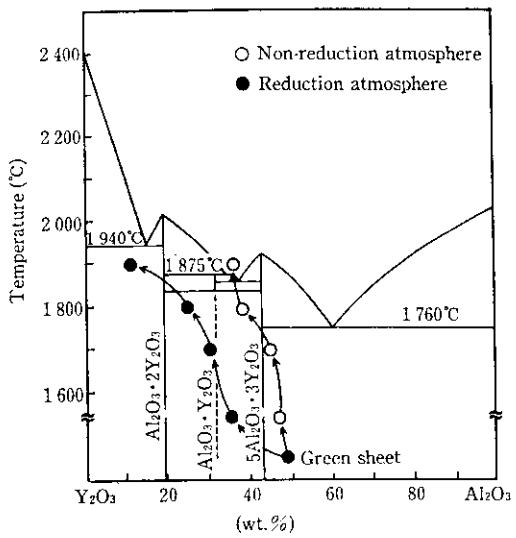

Fig. 6 Phase diagram of the system Al_2O_3-Y_2O_3 (Plots represent changes in calculated grain boundary composition based on Eq. (2) as a function of sintering temperature)

oxygen is all in the form of Al_2O_3 and Y_2O_3, the Al_2O_3 content was determined from Eq. (2).

$$Al_2O_3 \text{ content} = \frac{102}{48} \times ([\text{analytical value of O}] - [\text{O content of } Y_2O_3]) \cdots\cdots (2)$$

Figure 6 shows the changes in chemical composition of the liquid-phase at the grain boundaries in the sintering process plotted on the phase diagram of the system Al_2O_3-Y_2O_3[10]. In the nonreducing atmosphere, the compositions of conjugated oxides showed only small variations and could exist as liquid phases. In the reducing atmosphere, however, the proportion of Y_2O_3 increased due to the vaporization of oxygen and, therefore, no liquid phase was formed. This seemed to hinder densification.

As mentioned above, it is important to produce appropriate and stable atmospheric conditions because the furnace atmosphere strongly influences the sintering mechanism when liquid phases take part in the sintering.

5 Increasing Thermal Conductivity of AlN Sintered Bodies

As described in Chapter 2, techniques for reducing oxygen dissolved in the grains, grain-boundary precipitates, pores and grain-boundary voids, i.e. techniques for controlling macro- and microstructures, are necessary for obtaining AlN sintered bodies of high thermal conductivity with high reproducibility. These techniques are described in the following by way of examples.

The relationship between the oxygen content and

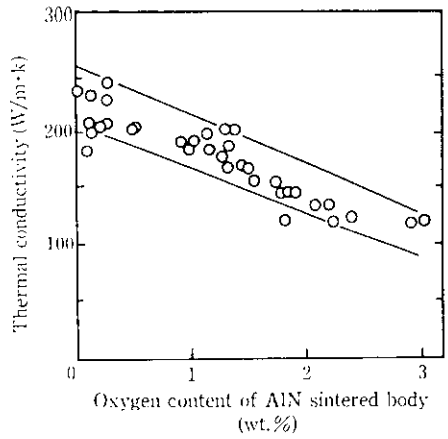

Fig. 7 Relation between oxygen content and thermal conductivity of AlN sintered bodies

thermal conductivity of AlN sintered bodies is shown in **Fig. 7**. The lower the oxygen content of AlN sintered bodies, the higher the thermal conductivity. When the oxygen content is high, the thermal conductivity decreases due to an increase in the phonon scattering[11] caused by the dissolution of oxygen into AlN lattices and due to grain-boundary precipitates with low thermal conductivity[12]. Therefore, the following measures are suitable for increasing the thermal conductivity of AlN sintered bodies:

(1) Adopt manufacturing process with a minimal oxygen pick-up.

(2) In the sintering process, the oxygen necessary for liquid-phase sintering is prevented from being released until densification is completed, and the oxygen content is lowered after that.

Sintering additives, as represented by Y_2O_3 and CaO, lower the oxygen concentration in the AlN grains[13] by causing the oxygen atoms in the AlN grain to gather at the grain boundary, and serve to suppress the scattering of phonons. Reducing additives such as CaC_2, are considered to perform an important role in lowering the oxygen content during sintering,[14] thus it is important to select an appropriate additive.

Mass production techniques were established by optimizing the selection of powder, additive, binder burnout and sintering conditions, etc.

The relationship between the microstructure and thermal conductivity of sintered bodies thus obtained is described in the following. **Photo 3** shows the microstructure obtained when the process was optimized. The grain size is almost uniform, there are no pores, and the grain-boundary phase is uniformly distributed. The thermal conductivity was high at 180 to 220 W/m·K. **Photo 4** shows an example in which defects with microvoids are present at the grain boundaries, although the grain size is almost uniform. The thermal conductivity was somewhat low at 160 W/m·K.

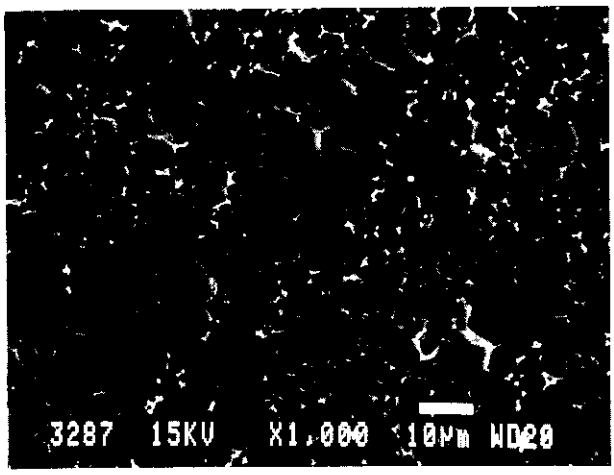

Photo 3 SEM micrograph of AlN (thermal conductivity 190 W/m·K)

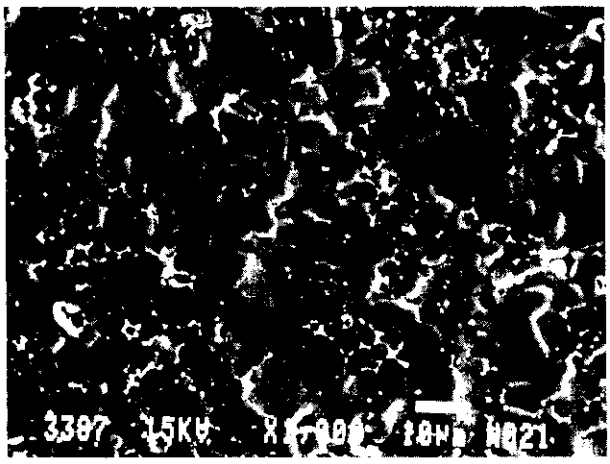

Photo 4 SEM micrograph of AlN: Grain boundary defect (thermal conductivity 160 W/m·K)

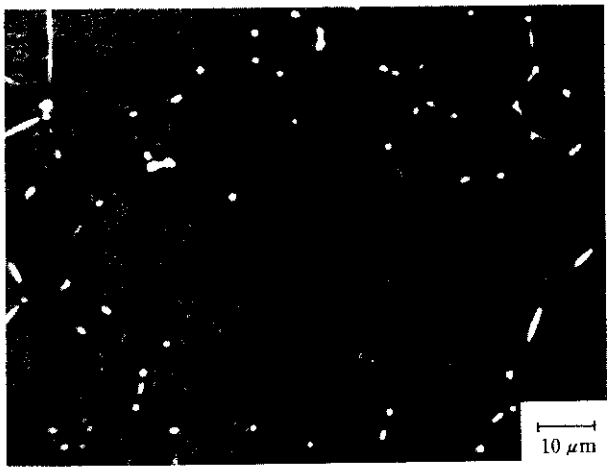

Photo 5 SEM micrograph of AlN: Thin grain boundary phase and large grain size (thermal conductivity 240 W/m·K)

Photo 5 shows the microstructure of a sintered body produced according to a special sintering method[15]. In this sintered body, only a small amount of a grain-boundary phase exists and the grain size is large. The thermal conductivity was 240 W/m·K.

As is apparent from the foregoing, thermal conductivity, which is the most important characteristic, is sensitive to microstructure. With what reproducibility thermal conductivity can be controlled was the key point of the subject development.

The properties of the developed AlN substrate with high thermal conductivity are given in **Table 2**, and the thermal conductivity distribution is shown in **Fig. 8**. A substrate with high thermal conductivity was obtained with high reproducibility. The sintering method was thought out in various ways to obtain a flat and smooth substrate with limited surface roughness and warping in the as-fired condition. The developed substrate has enjoyed a good reputation among users for use as metallized substrated by thick film printing and in other applications including copper plate bonding substrate.

Table 2 Properties of developed AlN substrate

Items	Properties	
Thermal conductivity	180~220	W/m·K
Electrical resistivity (RT)	$>10^{14}$	Ω·cm
Thermal expansion coefficient (RT~200°C)	3.9×10^{-6}	/K
Dielectric constant (ε) 1 MHz	8.9	
Dielectric loss (tan δ) 1 MHz	4.9×10^{-6}	
Insulation voltage	>30	kV/mm
Density	3.30	g/cm³
Modulus of rupture	45	kgf/mm²
Surface roughness (Ra)	<0.5	μm
Warping	<0.1/50 mm	mm

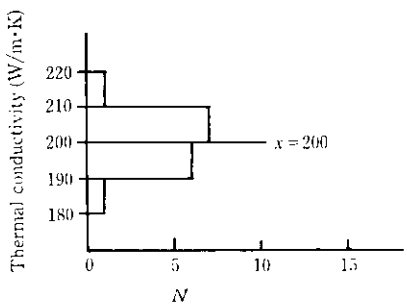

Fig. 8 Thermal conductivity distribution of developed AlN substrate

6 Metallization of AlN Substrates

6.1 Metallizing Techniques Necessary for AlN

Metallizing methods for forming circuits on AlN substrates, which are being developed or in the stage of practical applications, include, as for alumina substrates, the thick-film printing method, copper plate bonding method, high-temperature bonding method, thin-film forming method by physical vapor deposition, and electroless plating method.

The Thick-film printing method is the most widespread method for HIC, using the three types of pastes: a conductive paste comprising metallic powder of Au, Pt, Pd, Ag, etc.; a resistor paste consisting mainly of conductive oxide powder; a dielectric paste consisting mainly of glass powder. These pastes are successively screen printed to form a circuit on the substrate, followed by the firing process in each print process. This method is low in cost, suitable for mass production, and can form fine patterns.

In the copper plate bonding method, copper foil is directly bonded to the substrate like a printed circuit board. Although it is difficult to form fine patterns, high thermal conductivity can be obtained. This method has been increasingly applied, especially for large current use as in power transistors and thyristors.

In the high-temperature bonding method, powdered high-melting-point metals, such as Mo-Mn or W, were pasted, directly printed on a green sheet of AlN, and co-fired. In spite of the disadvantage of a high line resistance, this metallizing method is effective for making a multilayer substrate with wiring between layers.

The physical vapor deposition method involves forming conductor and resistor films of 1 μm or less on a substrate whose surface is smooth. Very fine patterns can be formed by using this method.

In the electroless plating method, a metallic film of copper or nickel is precipitated using a resist mask, following surface treatment of the substrate.

In all of the above methods, the bonding mechanism to a substrate of AlN, is different from that to a substrate of alumina. To conduct metallization which ensures high strength and reliability, it is necessary to develop techniques specially suited to AlN substrates.[16]

6.2 Thick-Film Metallization

This section describes the present state and problems of metallization techniques, giving the thick film printing method as an example.

6.2.1 Formation of thick-film conductor

It is generally more difficult to form thick-film conductors with AlN than with alumina.[17] When a paste for alumina substrates is used in AlN substrates, without modification, it is impossible to obtain a fired film of high adhesive strength for the following reasons:

(1) Since AlN is a nitride, its wettability with glass is different from that of alumina which is an oxide.[18]
(2) The thermal expansion coefficient of AlN is almost twice that of Al_2O_3. In glass, adjusted and optimized for Al_2O_3, thermal stresses are generated due to the mismatch of thermal expansion coefficients, and defects are liekly to form at the boundaries.
(3) Pastes suitable for alumina generally contain a large amount of Bi_2O_3, which reacts with AlN during firing to generate bubbles.

In recent years, however, conductive pastes that can be used in AlN have been developed by paste makers and several kinds are now commercially available. An experiment was conducted using three kinds of these pastes; the mix bond type (Paste A), the frit type (Paste B), and the direct bond type (Paste C).

These pastes were fired and sheet resistance, solder wettability, solder leach and adhesive strength were measured. The results of the measurements are shown in **Table 3**. An aging test was also conducted, and the results are shown in **Fig. 9**. From these results, it is judged that both solder characteristics and adhesive strength approach the levels required for practical application although the decrease in strength after aging poses a problem.

6.2.2 Formation of thick-film resistor

As for the conductor paste, a filler, glass, and vehicle are mixed to make the resistor paste, however, the glass content of the resistor paste is high compared to the conductor paste.

In general resistor pastes for alumina, a glass with a high bismuth content is used to increase wettability. When this glass is used in AlN, it reacts with AlN at

Table 3 Characteristics of think film conductor fired on AlN substrates

	Sheet resistivity (mΩ/□)	Solder leach* (cycle)	Solder Wet**	Adhesion strength (kg/4 mm²) Ave	Min	Max
PASTE A	18.0	4	>96%	3.70	2.39	5.00
PASTE B	4.1	1	>95%	3.43	1.05	5.47
PASTE C	15.6	0	>96%	1.03	0.70	1.81

* 290°C Sn60-Pb40 solder dipping cycles

** Coverage after 230°C Sn60-Pb38-Ag2 solder dipping

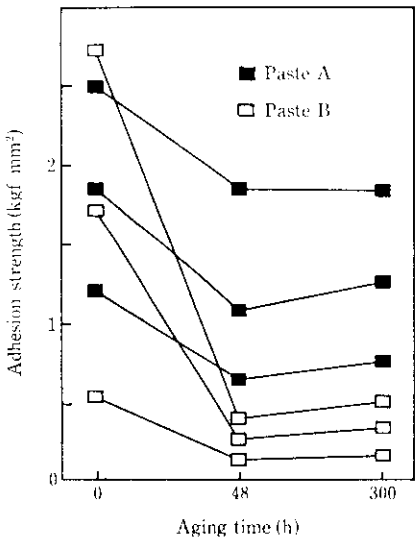

Fig. 9 Aging effect on adhesion strength of conductive thick film

temperatures near the firing temperature and blisters are formed due to the generation of N_2 gas.[19] A thick film of resistor with blisters results in fatal defects such as variations in resistivity and open lines. This phenomenon is especially serious in resistor pastes with a high glass content.[20]

To solve this problem, devitrifiable glass of low reactivity can be used.[19] Conventional pastes can also be used by oxidizing the surface of the AlN substrate[20].

Resistor patterns were printed using a resistor paste of the devitrifiable glass type (trial product) and the reproducibility of resistivity, thermal coefficient were measured. The results of these measurements are shown in **Table 4**. Resistors of less than 10 kΩ/□ were used. Although variations in resistivity, due to the aspect ratio of resistor patterns, are somewhat high, the reproducibility and thermal coefficient are good. Therefore, thick film resistors will be suitable for practical application.

The reproducibility and thermal coefficient were not good at higher resistivity values and when other pastes were used.

6.2.3 Prospects for thick-film printing method

From the foregoing, it was determined that the thick-film printing method enables the manufacture of circuits which can be practically applied, although there is still room for improvement in both the conductor and the resistor.

AlN metallization by thick-film printing will be further developed in two major directions in the future. First, there will be the development of conductive pastes of higher reliability and higher strength, as well as resistor pastes with good reproducibility of low resistivity and good thermal coefficients. Development of pastes for AlN metallization, which was considered difficult, is gradually progressing and has reached the level of practical application. If substrates can be directly metallized, costs will be reduced and thermal conductivity will not decrease.

Secondly, surface oxidization may enable the use of unmodified conventional pastes for alumina.[18,20] Studies on surface treatment techniques for this purpose are being actively conducted. If these techniques are put into practical use, it will become unnecessary to change operational conditions such as those in printing and firing.

7 Features and Applications of AlN Substrate

The features of the AlN substrate developed by Kawasaki Steel are summarized as follows:

(1) High thermal conductivity can be achieved with high reproducibility. The thermal conductivity is more than 180 W/m·K with ordinary firing and more than 240 W/m·K with special firing.

(2) The thermal expansion coefficient is 3.9×10^{-6}/K, which is close to that of Si (3.5×10^{-6}/K). It is possible to mount large-size semiconductor devices on the AlN substrate.

(3) AlN substrates have other electrical and mechanical properties equal or superior to those of alumina substrates.

AlN has already been used in the following fields, displaying superior heat release properties and a thermal expansion close to that of Si:

(1) Thermal release chips for laser diodes

Table 4 Characteristics of thick film resistor fired on AlN substrates

	Resistance (Ω)				Low TCR −55~25°C (ppm/K)	High TCR 25~125°C (ppm/K)
	1 mm×0.5 mm	1 mm×1 mm	1 mm×2 mm	1 mm×4 mm		
10 Ω/□	4.7±0.2	13.7±1.1	33.9±1.9	76.1±1.7	181	181
100 Ω/□	20.5±0.7	74.6±3.7	206.2±6.5	521.8±5.2	18	36
1 kΩ/□	240.1±6.2	1 093±30	33.9±1.9	76.1±1.7	29	27
10 kΩ/□	2 624±240	11 850±330	41 490±140	112 700±8 900	−99	−97

(2) Heat sinks for inverters and thyristors
(3) Substrates for high-frequency power transistors

To ensure that the market for AlN substrates will be extended further, it is important that AlN substrates should be put into practical use as highly integrated mounting substrates, as thick-film HIC substrates, and in the packaging field, especially for high-frequency IC.

8 Conclusions

Kawasaki Steel has developed techniques for AlN metallization and for massproducing AlN sintered bodies having high thermal conductivity and excellent IC substrate properties with good reproducibility.

(1) The company established techniques for manufacturing substrates having a thermal conductivity of more than 180 W/m·K, with high reproducibility, by developing high-purity fine AlN powder, a good organic binder, and binder burn-out and firing techniques.
(2) The following points are especially important for achieving high thermal conductivity:
  (a) High-purity, low-oxygen AlN powder is used. Impurities are further decreased in the substrate making process.
  (b) The grain growth in the sintering process is accelerated.
  (c) Pores in the sintered body are completely removed.
(3) Based on the metallization techniques (the thick-film method, the thin-film method, etc.) developed for alumina substrates, the company developed metallizing techniques suitable for AlN substrates, part of which have reached the stage of practical application.

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