

Thick film heater for aluminum nitride ceramics

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Abstract

Heater pastes for aluminum nitride (AlN) are not in common use at present. Therefore, heater pastes with AgPd conductive components are under development. This study shows the influence of the AgPd ratio in heater pastes on the film properties as well as on the heater characteristics. Therefore, three pastes with different AgPd ratios are prepared and tested. The formations of the heater materials are analyzed by insitu resistance measurements, X-ray studies and FESEM. The characterization of the heaters under electrical load and at different temperatures are investigated.

Key words: aluminum nitride, heater, thick film

Introduction

In the recent years aluminum nitride (AlN) became more important as substrate material in microelectronic components. Compared to other materials like aluminum oxide (Al_2O_3) and low temperature cofired ceramics (LTCC), AlN has an excellent thermal conductivity of more than 150 W/mK and very good mechanical properties as well as a good thermal shock resistance. Those properties make AlN an interesting material for heater applications. At present, thick film heaters are mostly realized on isolated steel substrates and the paste systems are commercially available. Those systems consist of a dielectric paste, heater pastes with different resistivities and contact paste. The dielectric film needs high isolation resistivity and breakdown voltage. At higher temperatures especially the breakdown voltage is decreased. The good isolating properties at higher temperatures are an important advantage of AlN.

A heater paste system based on Ag, Pd and lead free glass with thermal expansion coefficient (TEC) near that of AlN is under development. The influence of the AgPd ratio on the sintering behavior and the thermal coefficient of the resistance (TCR) is investigated. Furthermore, the heater structures are characterized under electrical load. Their temperature is measured by an IR camera.

Experimental Procedure

Test pattern for heater characteristics

The heater properties were determined with a test structure, shown in Figure 1.

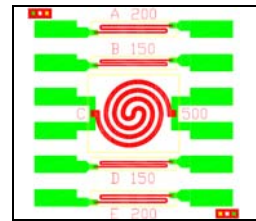


Figure 1: Test structure for heater measurements

The heater layout includes three heater structures with different line widths. For the test the coil structure with a line width of 500 μm and a length of 57 mm was used.

Table 1: Screen parameters

Structure	Mesh	Film	Paste
Heater	325	25 μm	A, B, C
Contact pads	200	25 μm	FK 1220
Overglaze	200	25 μm	FK 4027

To realize this test structure it is necessary to print three screens: the heater structure, the contact pads and the overglaze (Table 1).

Each film is separately fired, the heater and the contact pads at 850°C, and the overglaze at 650°C.

In situ resistance measurements

The dried films were contacted with Pt wires and then fired in a furnace. The heating and cooling rate was 10 K/min, and the peak temperature of 850°C was held for ten minutes.

Resistivity and its thermal coefficient (TCR)

The electric resistance was determined by four-point measurement. The sheet resistivity was calculated for a dried thickness of 21 µm. The TCR was determined between 30°C and 600°C.

Field emission scanning electron microscope (FESEM)

The surface was characterized by FESEM, using a Digital Scanning Microscope 982 (company LEO) at 2 kV.

X-ray diffraction measurements

X-ray diffraction measurements were performed with a XRD 7 diffractometer (company Siefert). The diffractometer works with Cu K α radiation and Theta/2Theta geometry.

Electrothermic continuous load

The heaters were characterized by a continuous electric load. The test samples were prepared by soldering wires on the contacting pads. During the measurement the samples are connected to an aluminum cooling block. An Ag-containing heat-conducting paste was used for a good thermal connection. The test voltage was determined for every sample for heater temperatures at about 200°C and 400°C. The temperature and the resistance were measured over the time. The peak temperature of the heater was determined by IR camera measurements.

Results and Discussion

Film formation

Three pastes were prepared with different AgPd ratios (Table 2). The formation of the dried films during the firing process was investigated by insitu measurements of the resistance. Furthermore, XRD measurements were carried out at samples fired at different temperatures. The varied AgPd ratios influence the sintering behavior of the films as is shown by the insitu resistance measurements (Figure 2).

Table 2: Paste composition

Paste	AgPd ratio	Pd [vol%]
A (model paste)	1 : 23	54.6
B	1 : 1	45
C	10 : 1	4.5

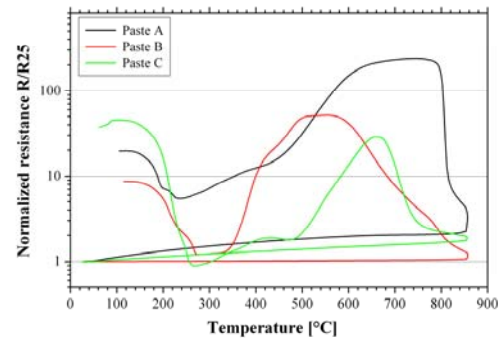
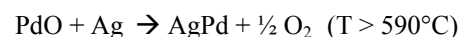
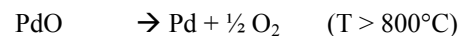


Figure 2: In situ resistance measurements

The change of resistance can be arranged in different ranges.

- (I) In the first temperature range (25...250 to 300°C) the polymer of the organic vehicle is burnt out. After drying the metallic particles were separated by the polymer. In the burn out process the contacts between the metallic particles were generated accompanied by a decrease in resistance. For all three films a minimum of resistance is measured. The resistance in the minimum between 200 and 300°C increases with the palladium content in the film.
- (II) At temperatures higher than 230°C the palladium powder begins to oxidize and the resistance is increased. The highest resistance was measured for the film with the lowest AgPd ratio. By increasing the Ag content a competing reaction, namely the formation of AgPd alloy is observed. The decomposition of the PdO and the AgPd alloy formation cause the decrease of resistance.
- (III) During the cool off range the resistance is decreased in dependence of the AgPd ratio. The temperature coefficient of the resistance is measured.

Thermogravimetric analyses of Pd powder and a mixture of Ag and Pd powder (AgPd ratio: 1/1) are shown in Figure 3 [1] These curves demonstrate the above mentioned reactions.



The palladium powder is oxidized at temperatures higher than 230°C and is decomposed at 800°C. In the presence of Ag (AgPd ratio: 1/1) the decomposition temperature is reduced up to 590°C. At temperatures smaller than 800°C the formed AgPd alloy is more thermodynamically stable than the PdO.

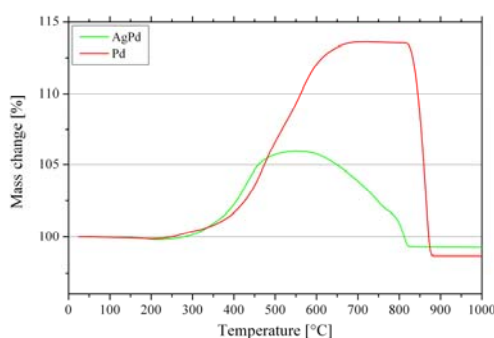


Figure 3: Thermogravimetric measurement (air at 10K/min)

The X-ray measurements of the different films in three states dried and fired at 500 and 850°C are illustrated in Figures 4 to 6. In all dried films Ag, Pd and ZnAl_2O_4 (paste A and C) are identified. The samples prepared with the paste A and fired at 500 and 850°C show Pd and PdO. Ag is not detected anymore. It is alloyed with Pd. After firing the peak of Pd is smaller caused by the growth of the Pd grains.

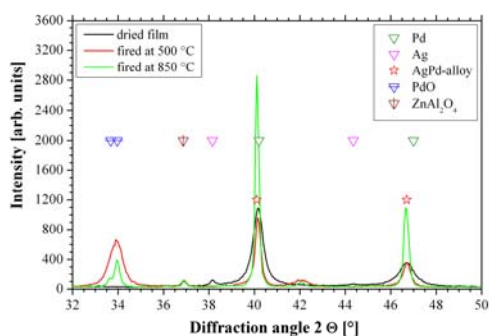


Figure 4: X-ray of sample A

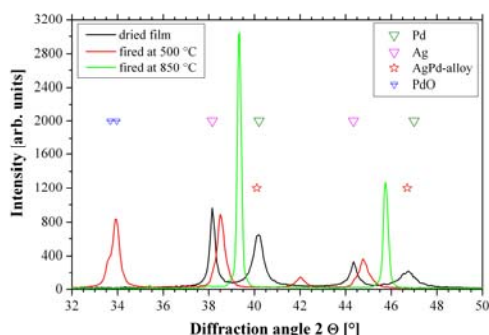


Figure 5: X-ray of sample B

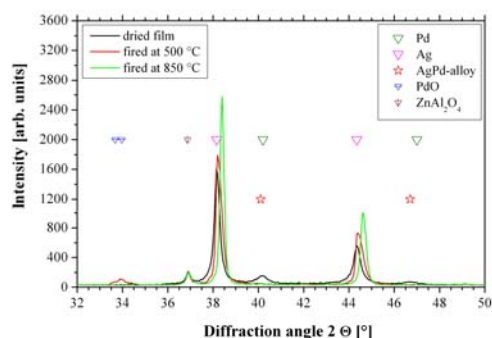


Figure 6: X-ray of sample C

The formation of the AgPd alloy can clearly be indicated for paste B, on account of the same amount of Ag and Pd in the dried film. At 500°C Pd can not be detected anymore, but PdO is found as well as the shift of the Ag peak to higher angles (lattice spacing). This indicates that both processes, oxidation and alloy forming, occur in the same time. At 850°C PdO is not found. The alloy formation is finished, shown by a further shift of the Ag peak. For the samples prepared with paste C the same phases are found. But the intensity of the Pd and the PdO peaks are lower caused by the smaller amount of Pd in the film.

Figure 7 shows FESEM images for the different films in the three states. The images of the dried films show glass, Ag and Pd particles. The growth of the Pd and Ag grains can be observed after firing at 500°C. The film with the highest AgPd ratio shows the beginning of the sintering of the Ag powder. At 850°C the formation of the AgPd alloy of paste A is not completed. The pictures of the films prepared with paste B and C and fired at 850°C show the grain growth and the complete formation of the alloy.

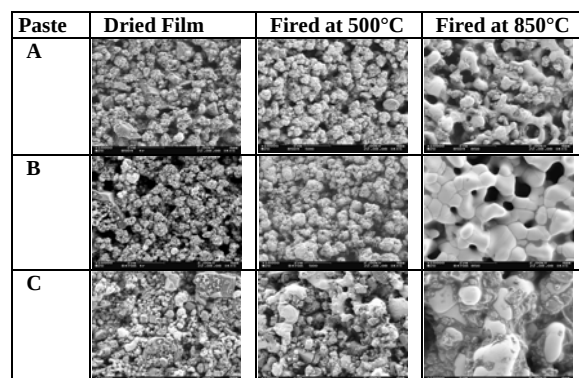


Figure 7: FESEM images for different sintering states

Resistivity and TCR of the fired film

Table 3 shows the sheet resistivity and the calculated TCRs of the fired films. The TCR depends on the AgPd ratio. The lowest TCR (around $50 \cdot 10^{-6}/\text{K}$) can be achieved by mixing the metal powders one to one. To get higher TCRs it is necessary to increase the amount of one of the metals.

Table 3: Resistivity and TCR

Paste	Rsq	TCR [$10^{-6}/\text{K}$]
A	99	1720
B	100	48
C	133	857

Figure 8 shows the curve shape of the normalized resistance to the temperature.

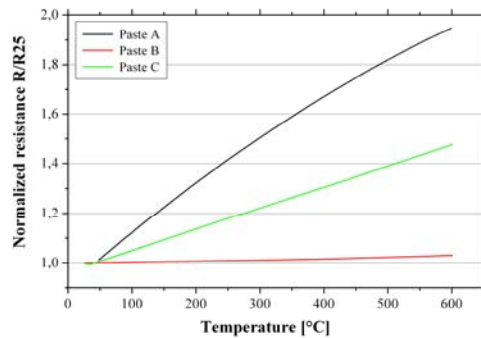


Figure 8: R-T curve

The curves were fitted by a linear function:

$$y = a + b \cdot x,$$

where a is the point of intersection and b is the slope. The linear fit calculates a correlation coefficient R , which indicates the quality of the fit. Table 4 shows the results of the linear fit for the three pastes.

Table 4: Fitting parameters

Paste	a	b	R
A	0.9423	$1720 \cdot 10^{-6}$	0.99767
B	0.9980	$48.4 \cdot 10^{-6}$	0.99312
C	0.9725	$857 \cdot 10^{-6}$	0.99999

The correlation coefficient is nearly 1 for all pastes and has the best value for paste C. This indicates a very good linearity of the TCR, whereas for paste A and B a parabolic respective cubic function gives a better fit. The calculated slopes reflect the TCR of the pastes.

Heater characterization

The heaters were investigated by electrothermic continuous load measurements. By applying about

140 W the samples heat to 180-200°C, whereas the load of 250 W results in 360-440°C. Because of the constant voltage supply, any resistance change results in small power and hence temperature shift.

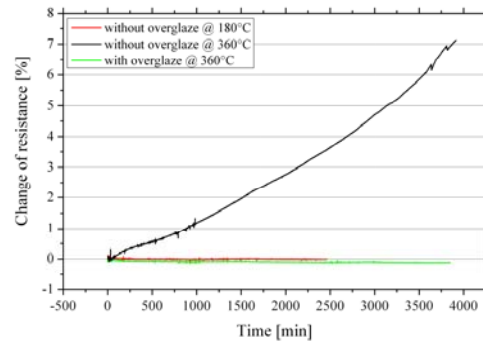


Figure 9: Heater test by continuous load (heater B)

The results for heater B are shown in Figure 9.

The heater without overglaze was heated to about 200°C and 400°C. This means the temperature of the hottest point of the heater. For the lower temperature the resistance is changed $<0.1\%$ over the time. At 360°C the resistance increased with loading time up to 7%. The reason for this aging could be the fusing of conductive paths or oxidation of Pd. The overglazed heater shows only a small change of resistance of 0.1%. The temperature distribution of the loaded heater is shown in Figure 10.

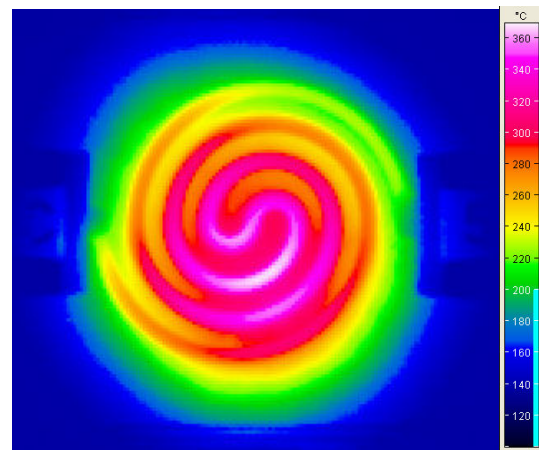


Figure 10: IR pattern of loaded heater B with overglaze, after 64 h

X-ray measurements were done for thermally aged samples with and without overglaze at 200°C and 400°C. This measurement (Figure 11) shows that PdO peaks at around 33.9° occur only after the aging at 400°C without overglaze. This confirms the assumption of the PdO forming on the unglazed heater.

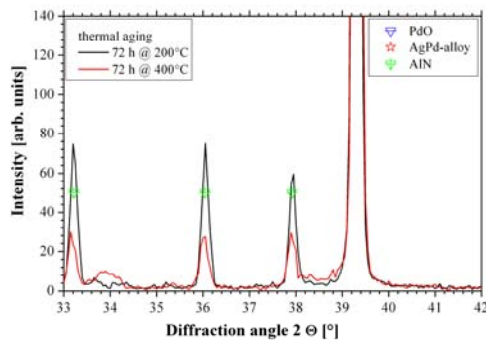


Figure 11: X-ray of the thermally aged film (heater B)

Figure 12 shows the results of the electrothermic loading for heater C. The loading was done with a unglazed heater at 200°C and 440°C. Because of the low Pd content a stable AgPd alloy is formed and no oxidation occurred during the loading. X-ray measurements confirm this result. The diffractogram in Figure 13 shows no PdO peak at 33.9 °.

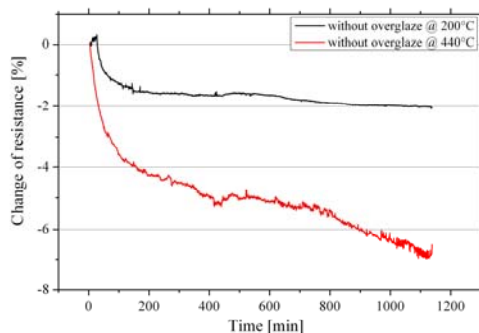


Figure 12: Heater test by continuous load (heater C)

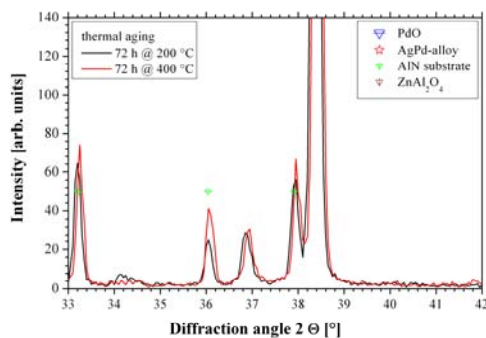


Figure 13: X-ray of the thermally aged film (heater C)

However, the resistance decreased during the loading time. The decrease is stronger for the higher temperature (~7 %) compared to the lower

temperature (~2 %). This behavior can be explained by the sintering of the Ag film.

Conclusion

AlN substrates can be an important substrate material for heater applications. The paper presents some results of the development for thick film based heater pastes. In this work pastes with different AgPd ratios were used. Various test samples were fabricated and examined using different methods like thermogravimetric measurements, X-ray diffraction measurements and field emission scanning electron microscopy. The electric and the thermal performance of these pastes were also discussed.

References

- [1] C. Kretzschmar, P. Otschik (1998): Thick Film Alloy Resistor System on AlN, 43rd International Scientific Colloquium, Technical University of Ilmenau, September 21-24