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Influence of temperature and glass composition on aluminum nitride contact angle

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Abstract. Results of research of different glass compositions for possibility of their application in metallization pastes intended for ceramics based on aluminum nitride are presented in this article. It includes research of contact angle of aluminum nitride with glasses of different compositions at different temperatures and different roughness of ceramics.

1. Introduction

Ceramics based on aluminum nitride is a perspective material used in electronics as housings and substrates for electronic chips due to its unique combination of high thermal conductivity and high dielectric properties. More than 90% of aluminum nitride ceramics market are presented by metallized ceramic products, therefore it is necessary to develop metallization pastes intended for ceramics based on aluminum nitride. At the moment the process of aluminum nitride metallization is poorly understood. One of the most effective ways of ceramics metallization is using glass as binding additive in metallization pastes.

In accordance with world trends of development, one of demanded market segments is electronics and electrical engineering (ceramic elements for electronic industry, including thermal loaded elements of semiconductor devices, high-power LEDs; insulators for various applications for electrical engineering and energetics). One of promising directions is producing high thermal conductive materials and products based on aluminum nitride. Aluminum nitride possesses a row of unique properties compared with other materials used in microelectronics [1]. Aluminum nitride application as base in high-power LEDs manufacturing requires applying a conductive paste on it. Conductive low-temperature silver-containing pastes developed in a large amount are intended for oxide materials, so they have low adhesion to aluminum nitride. It is possible to increase adhesion of metallization paste to aluminum nitride by introduction glass binder to their composition [2], which must wet aluminum nitride at low temperatures.

The aim of this work is development of glass additive which wets aluminum nitride at temperatures up to 1000 °C.

2. Experiment

Samples in the form of aluminum nitride discs sintered at relative density 98% were used in the work. The discs were subjected to grinding and cleaning. Surfaces were cleaned from organic pollutants by isopropyl alcohol and dehydrated by annealing at temperature 600 °C. To determine contact angle an installation has been constructed, a schematic diagram is shown in Figure 1.

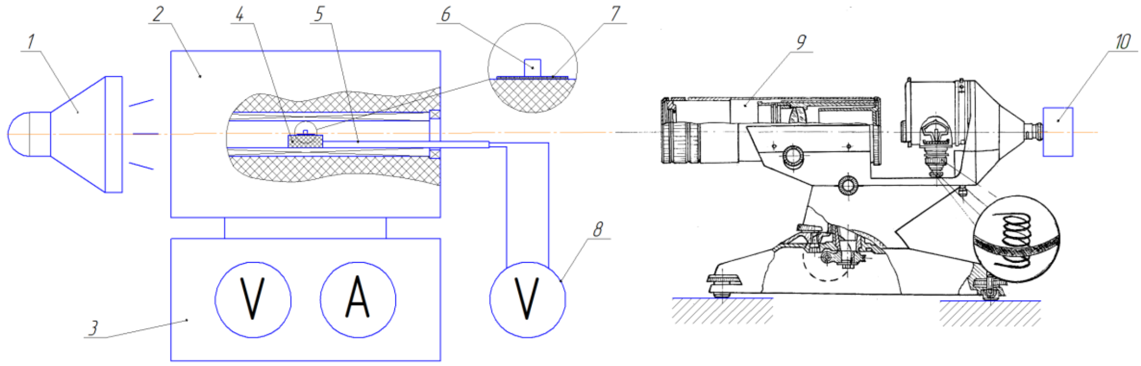

Figure 1. The installation schematic diagram

The installation consists of: 1 –lighting system; 2 –tubular furnace at a controlled heating rate; 3 – heating control system; 4 – sample stand; 5, 8 – thermocouples for the temperature control of the sample; 6 – glass sample; 7 – substrate (disc) of AlN; 9 – optical system; 10 – digital camera with an image displayed on the computer display.

The investigated glass was pressed in the form of cylinder with 3 mm diameter and 5-6 mm tall. In the pressing process does not use organic binders. After setting sample the furnace was heated with rate from 7 to 10 °C/min. When the temperature reached 600 °C the sample began to be photographed every 10 °C. Image processing was carried out using the software «Micro-Analiz Pro» supplied with the digital camera.

For the work were selected glass compositions in the system B_2O_3-SiO_2-Na_2O, which provide low cooking and softening temperatures. The compositions are shown in the Table 1.

Table 1. Ratio of components in the selected glasses.

Letter	Content of components, mol. %							Total
	Na_2O	CaO	B_2O_3	SiO_2	V_2O_5	K_2O	PbO	
a	20	5	10	65	–	–	–	100
b	20	5	10	55	10	–	–	100
c	10	5	10	65	–	10	–	100
d	20	5	10	55	–	–	10	100
e	10	5	10	75	–	–	–	100

Calculation of properties of the selected glasses was carried by specialized software SciGlass version 7.0. The one especially important of them is dependence of viscosity on temperature. Calculation results are shown in Figure 2.

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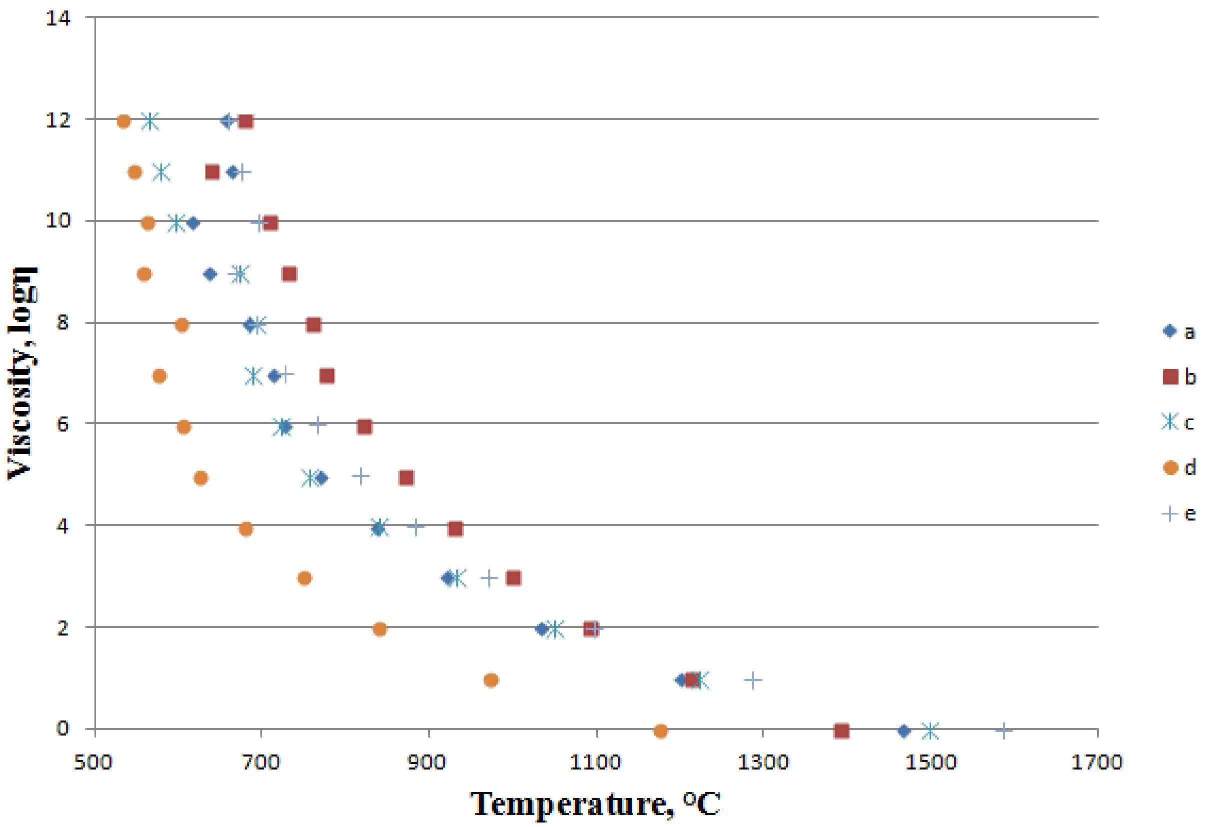


Figure 2. Dependence of viscosity on temperature

Due to calculated data one can see that the least refractory composition is composition "d" containing PbO, and the most refractory is composition "b" containing V_2O_5.
The glasses of proposed compositions were cooked whereupon they were grinded till full passing through the sieve with cell size 63 mkm. The glass powder was pressed into samples in the form of cylinder with diameter-to-height ratio equal 1/2. In Fig 3, 4 are shown sample photo at the different temperatures and data of contact angle definition.

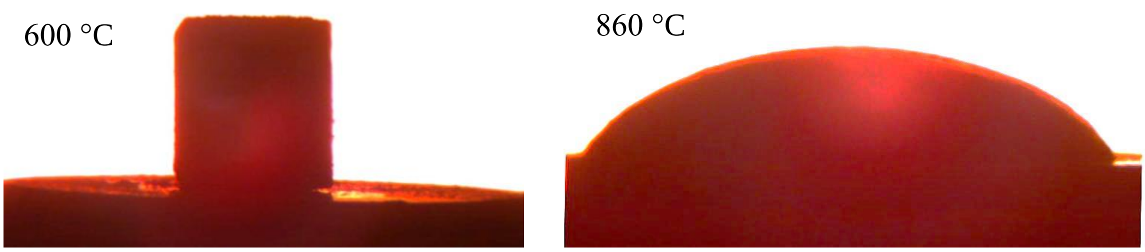
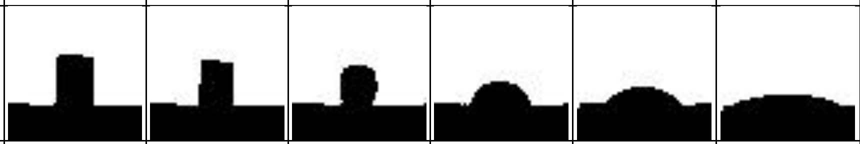
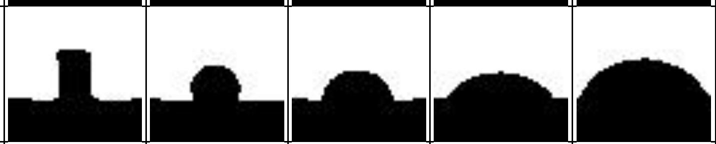
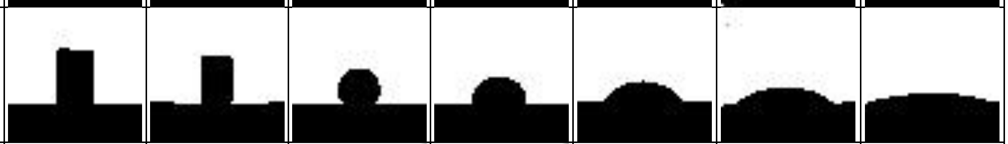
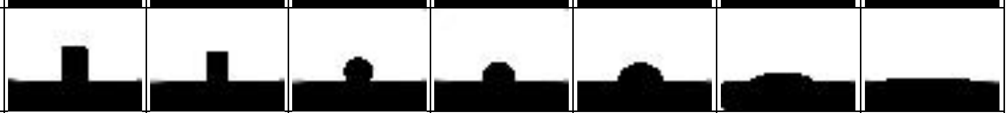
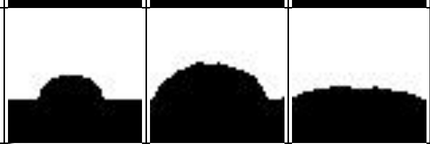
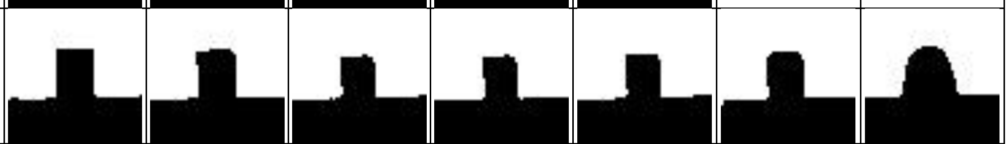


Figure 3. The glass of composition «d» samples photo at the temperatures 600 and 860 °C

Table 2. Glass samples images wetting polished and unpolished aluminum nitride samples at different temperatures.

		600	700	800	900	1000	1100	1200
a	polished							-
	unpolished							
b	polished						-	-
	unpolished							
c	polished							
	unpolished							
d	polished				-	-	-	-
	unpolished						-	-
e	polished							
	unpolished							

Images of glass samples at different temperatures on polished and unpolished aluminum nitride discs are shown in the Table 2. In this table one can trace process of glass spreading on the aluminum nitride substrate, as well as clearly see that in practically all cases the polished aluminum nitride samples are wetted by the glass better than unpolished. Apparently it's connected with the formation of air pockets reducing wettability on the surface of unpolished samples.

It is also noteworthy that the glass of composition "e" turned up the most refractory one contrary to calculated data.

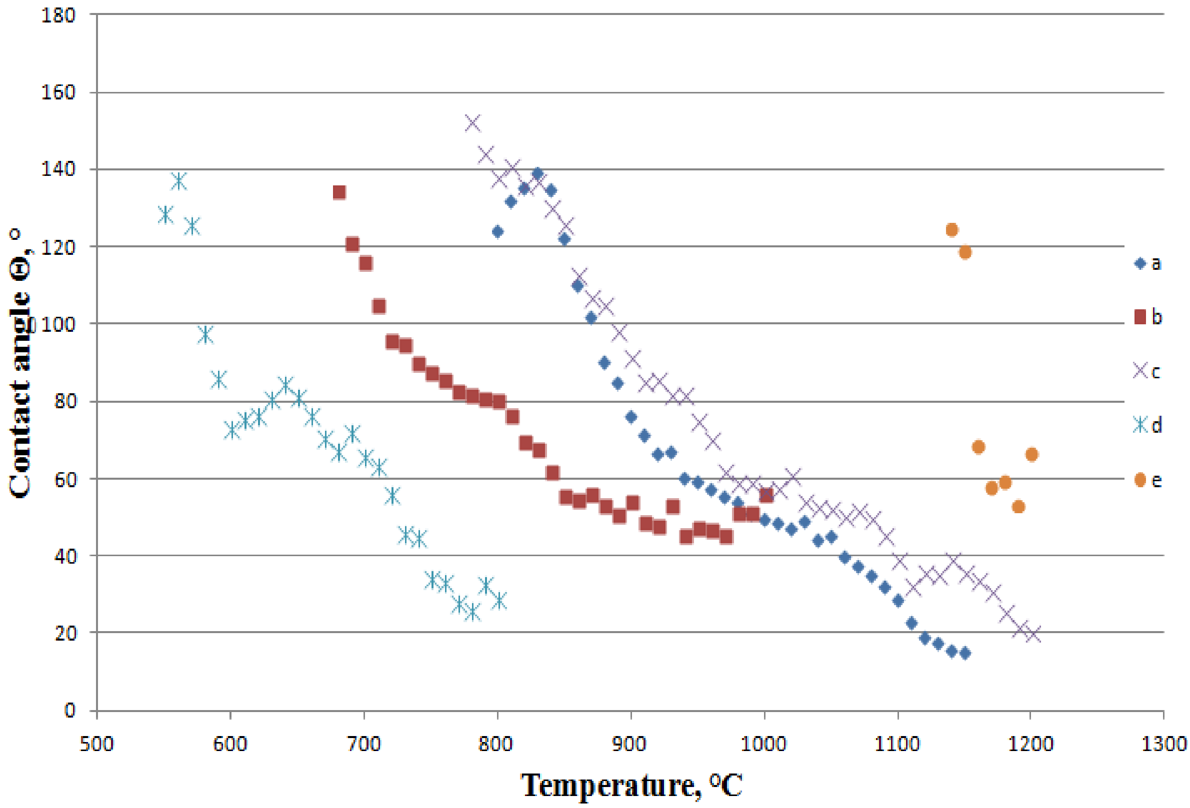


Figure 4. Dependence of aluminum nitride contact angle on temperature

Figure 4 shows diagrams of dependencies of contact angle of polished aluminum nitride samples with glass on temperature. One can see that these dependencies are similar in nature. Firstly, there is a maximum when the drop becomes spherical shape, then the drop begins to spread, and contact angle is decreasing. Secondly, there is an inflection associated with foaming glass: initially drop volume increases, then glass begins to be clarified and the foam is settling. Further, the contact angle goes to zero.

3. Conclusions

As one can see in experimental data shown in Table 2 and Figure 4, the glasses of compositions "d" and "b" respectively containing lead and vanadium begin to wet aluminum nitride at the lowest temperatures. These glasses can be used for metallization pastes based on silver which are burned at temperatures 800÷920 °C. The glass of composition "e" begins to wet aluminum nitride only at temperatures of about 1150 °C and in the future, possibly, it may be used in the molybdenum-manganese metallization pastes which are burned at temperature 1200÷1300 °C.

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