

**INFLUENCE OF DISPERSION OF THE BASE MIXTURE ON THE YIELD
OF ALUMINUM OXYNITRIDE**A.S. Shulzhenko

Scientific Supervisor: Ph.D. I.B. Revva

Tomsk Polytechnic University, Russia, Tomsk, Lenin str., 30, 634050

E-mail: alshs93@mail.ru**ВЛИЯНИЕ ДИСПЕРСНОСТИ ИСХОДНОЙ СМЕСИ
НА ВЫХОД ОКСИНИТРИДА АЛЮМИНИЯ**А.С. Шульженко

Научный руководитель: к.т.н. И.Б. Ревва

Национальный исследовательский Томский политехнический университет,

Россия, г.Томск, пр.Ленина 30, 634050

E-mail: alshs93@mail.ru

***Аннотация.** В работе исследовано влияние дисперсности исходных порошков оксида алюминия (Al_2O_3) и нитрида алюминия (AlN) на выход оксинитрида алюминия. Рассмотрена зависимость образования AlON от различных температур.*

Amorphous aluminum oxynitride (AlON) possesses unique properties of high dielectric strength, high resistivity, low loss, high decomposition temperature, chemical inertness, and high thermal conductivity. The main aim of the current research was to study the influence of dispersion of the initial mixture on the yield of AlON.

Aluminum oxynitride (AlON) ceramic material is a cubic solid solution of aluminum oxynitride. Its composition accords with the formula of $Al_{(64+x)/3} \square_{(8-x)/3} O_{32-x} N_x$, where \square is a cation vacancy. If the value of x equals 5, its formula is $Al_{23}O_{27}N_5$, the most stable phase of AlON. In view of the interesting optical, chemical and mechanical properties, aluminum oxynitride spinel (AlON) has potential applications as a high-performance structural ceramics and advanced refractory. In addition, it has been processed into fully dense transparent material and shows promising mechanical and optical properties suitable for use in infrared and visible window applications. AlON is a solid solution of Al_2O_3 and AlN. AlON is a solid solution in the Al_2O_3 - AlN pseudo-binary system. Its crystallographic structure is cubic spinel, thus its optical properties are isotropic. It is usually manufactured from powder mixtures of alumina and AlN. The previous investigation concluded that the γ -phase (AlON) is stable between 60 and 73 mol% Al_2O_3 for all temperatures between 1750°C and 2000°C. [1,2]

One of the main conditions for a more complete synthesis of the process is to acquire as high as possible contact area between the particles of raw materials. To achieve the full density of arrangement, we used alumina micron powder with aluminum nitride nanopowder (composition 1). To compare the effectiveness of micro- and nano-sized powder a mixture of powders of micron aluminum oxide and nitride was composed. (composition 2). Processing of optically transparent AlON is more difficult

«ПЕРСПЕКТИВЫ РАЗВИТИЯ ФУНДАМЕНТАЛЬНЫХ НАУК»

than the synthesis of opaque single phase AlON, because the material must be fully dense, pure, and free of any secondary phases. There are several methods of getting transparent ceramic: simple reaction of Al_2O_3 and AlN, carbothermal reduction of Al_2O_3 , plasma arc synthesis, and self-propagating high-temperature synthesis (SHS). [3] In our investigation we used the first method, which consists of some characteristic stages:

- 1) Mixing powders (ball milling in alcohol with ZrO_2 balls during 20 min. at the rate of 450 rotations per minute).
- 2) Drying the powder. (at 60°C for 4 hours.)
- 3) Filling a mold.
- 4) Pressing a blank to nearly net-shape and ~60% of the theoretical density (~20% oversize).
- 5) Binder burnout.
- 6) Sintering the blank at high temperature for an extended time (to allow atomic diffusion).

In this research was used Al₂O₃ Almatix (made in Germany), which is present white powder with a bulk density of 0.996 gm / cc. Determination of the phase composition of the starting powders and products synthesis was performed on the X-ray diffractometer DRON-3M. Samples of the starting materials previously crushed in an agate mortar to a particle size of 63 microns. According to X-ray diffraction analysis, the powder used is represented by a single phase Al₂O₃ (card number 000-11-06). The second major component used aluminum nitride AlN various dispersion. Pour density AlN-nano was 0.142 gm / cc and 0.776 AlN-micro gm / cc. According to X-ray analysis, both samples are composed of aluminum nitride single phase AlN.

To study the synthesis of oxynitride phase molded into tablets with a diameter of 30 mm and a height of 3 - 4 mm, the used compacting pressure of 7 tons. Polyvinyl butyral is used as the binder component in an amount providing sufficient strength of the molded tablets. The first firing is carried out at temperatures of 1750 and 1850 °C and held at maximum temperature for 3 hours. At the output of the first stage turned opaque, but strong enough tablets.

Results. During the firing process occurs baking samples, accompanied by a change, specifically the size reduction of the samples. Found that a composition 1 sintered more rapidly than the composition 2. In this case, at all temperatures firing linear shrinkage of composition 1 is 3 times more than shrinkage of composition 2. Using of the nanopowder in the composition 1 resulted in a more dense samples (Fig. 1).

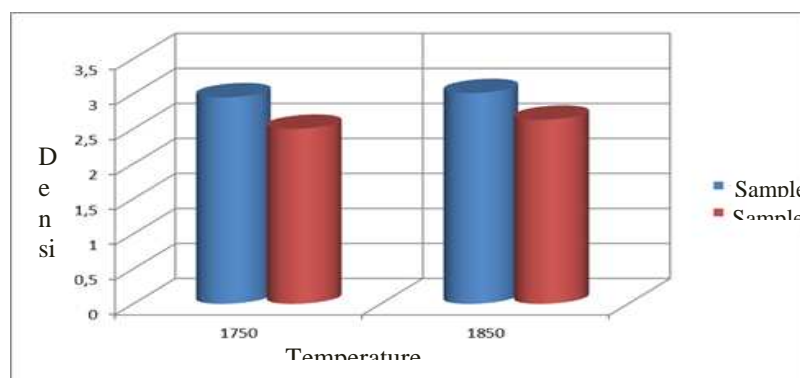


Fig. 1. Properties of the fired samples

XRD analysis of the test samples (Fig. 2, 3) showed that, at any temperature of firing there are peaks of the synthesized aluminum oxynitride and aluminum nitride in samples of various compositions.

And with increasing firing temperature intensity of the peaks of aluminum oxynitride $Al_{23}O_{27}N_5$ increases, which indicates an increase in the number of its synthesized blends. Found that the use of aluminum nitride nanopowder promotes the formation $Al_{23}O_{27}N_5$ at lower temperatures, as already at $1750\text{ }^{\circ}\text{C}$ for the main phase of the X-ray is aluminum oxynitride. In the case of a mixture of 2 (micron powders aluminum oxide and nitride) at a temperature of $1750\text{ }^{\circ}\text{C}$ firing on radiographs main phase is AlN.

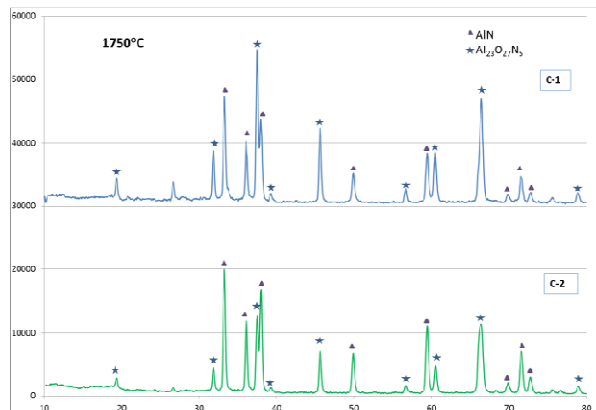


Fig. 2. Radiographs of samples fired at a temperature of $1750\text{ }^{\circ}\text{C}$

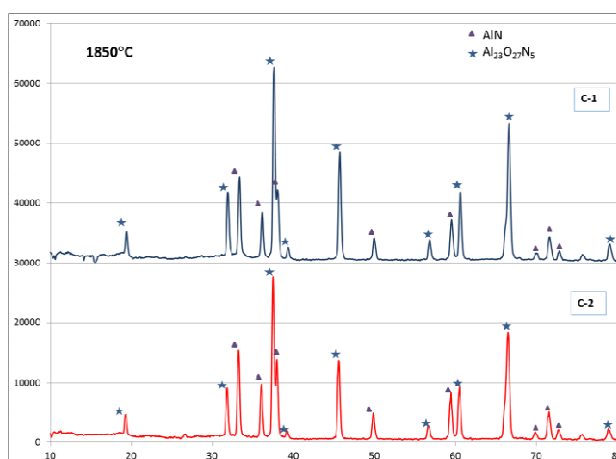


Fig. 3. Radiographs of samples fired at a temperature of $1850\text{ }^{\circ}\text{C}$

Increasing the synthesis temperature to $1850\text{ }^{\circ}\text{C}$ cause an intensification of the synthesis of oxynitride for the composition 1 and 2. That way, the work shows the efficiency of the use of powders with different particle sizes in the process of synthesis of aluminum oxynitride.

REFERENCES

1. James W. McCauley, Parimal Patel, Mingwei Chen, Gary Gilde, Elmar Strassburger, Bhasker Paliwal, K.T. Ramesh, Dattatraya P. Dandekar. AlON: A brief history of its emergence and evolution.// Journal of the European Ceramic Society. – 2009 – № 29 – P. 223–236
2. N. Zhang, B. Liang, X.Y. Wang, H.M. Kan, K.W. Zhu, X.J. Zhao. The pressureless sintering and mechanical properties of AlON ceramic// Materials Science and Engineering A. – 2011 – №28 – P. 61–63
3. S.F. Wang, J. Zhang, D.W. Luo, F. Gu, D.Y. Tang, Z.L. Dong, G.E.B. Tan, W.X. Que, T.S. Zhang, S. Li, L.B. Kong. Progress in Solid State Chemistry// “Transparent ceramics: Processing, materials and applications”. – 2013 – № 41 – p. 20 – 44