Evaluation of the method of measuring crack resistance by the introduction of the vickers indentor for aluminum oxynitride ceramics

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Abstract. Results are given after studies of materials on the aluminum oxynitride (AlON) for their surface crack resistance using. The comparison of fracture toughness data obtained by the two methods and two types of ceramics. Assessment of fracture toughness data in comparison with the results from the literature in the field of ceramic materials research.

1. Introduction

Oxinitride ceramics (AlON) is a very promising material today. AlON ceramics is used in many areas of technology: engineering, aviation, instrument making, etc. Ceramics of aluminum oxynitride has a combination of properties that are not inherent in one material, such as durability, transparency and low density.

Mechanical properties, such as hardness, density, fracture toughness and crack resistance are important for transparent ceramic materials used in protective structures. There are not many methods for evaluating these parameters of ceramic materials, especially taking into account the specifics of their destruction. Accordingly, one of the methods is to find the crack resistance parameter using a Vickers Indenter, since there is a relationship between the hardness and crack resistance of ceramic materials [1,2].

To determine the crack resistance, a pyramidal-shape indenter is introduced. Cracks extend from its corners. Such imprints are conventionally divided into two types according to the definition of Dr. Palmqvist [3] and Anstist [4] (figure 1). The nucleation of Palmqvist cracks occurs in the interface between plastic deformed elastic regions, but its nucleation point is located close to the sample surface in the diagonal direction of the indenter (figure 1, a). The model in (figure 1, a) is a Palmqvist model, where Palmqvist cracks are located on both sides of the pyramid’s imprint and internal lateral cracks extend from the top of the pyramid. The stress state on the surface is similar to that in the case of median crack nucleation, however, faults existing surface may be responsible for stress concentration points that enhance the nucleation of Palmqvist cracks. In model (figure 1, b) the crack has the shape of a circle around the indent, called a semicircular “half-penny”. Lengths parameters (a, c, l) are shown schematically in the figures. Accordingly, for these two models there are many options for mathematical interpretation (table 1.).
Table 1. Some equations developed for the determination of fracture toughness in brittle materials

| No. | Equation | Crack model       |
|-----|----------|-------------------|
| 1   | $K_{IC} = 0.016 \left( \frac{E}{H_V} \right)^{\frac{1}{2}} \frac{P}{c^\frac{3}{2}}$ | Half-penny        |
| 2   | $K_{IC} = 0.067H_\sigma^{\frac{1}{2}}\left( \frac{E}{H_V} \right)^{\frac{1}{2}} \left( \frac{c}{a} \right)^{\frac{3}{2}}$ | Half-penny        |
| 3   | $K_{IC} = 0.0752 \frac{P}{c^2}$ | Half-penny        |
| 4   | $K_{IC} = 0.0725 \frac{P}{c^2}$ | Half-penny        |
| 5   | $K_{IC} = 0.014 \left( \frac{E}{H_V} \right)^{\frac{1}{2}} \frac{P}{c^\frac{3}{2}}$ | Half-penny        |
| 6   | $K_{IC} = 0.0089 \left( \frac{E}{H_V} \right)^{\frac{1}{2}} \left( \frac{P}{\alpha c^3} \right)$ | Curve-fitting     |
| 7   | $K_{IC} = 0.0889 \left( \frac{H_VP}{\alpha c^3} \right)^{\frac{1}{3}}$ | Curve-fitting     |
| 8   | $K_{IC} = 0.4638 \left( \frac{E}{H_V} \right)^{\frac{1}{2}} \frac{P}{a^2 \times 10^6}$ | Curve-fitting     |
| 9   | $K_{IC} = 0.018 \left( \frac{E}{H_V} \right)^{\frac{1}{2}} \frac{P}{c^2}$ | Curve-fitting     |
| 10  | $K_{IC} = H_\sigma^{\frac{1}{2}}\left( \frac{E}{H_V} \right)^{\frac{1}{2}} \times 10^6$ | Curve-fitting     |
For example, Ivanov [5] applies equation 1 (table 1) to determine the hardness of aluminum oxide ceramics. In this paper, the crack resistance parameter may indicate a seal inside the sample and the “degree” of powders sintering.

2. Materials and techniques

Al₂O₃ and AlN powders obtained by plasma chemical synthesis were used as starting material for the synthesis of Al₂₃O₇N₅ ceramic samples [6,7,8]. The parameters of the powders are given in (table 2).

| Table 2. Characteristics of starting powders |
|---------------------------------------------|
| Initial powder   | Chemical purity, % | Average particle size, μm |
| Al₂O₃            | 98,0               | 0,1                       |
| AlN              | 98,0               | 10,0                      |

The powders were mixed in a planetary micromill in a ratio of 40:60 for 50-60 minutes. The process was carried out in isopropyl alcohol to prevent agglomeration of powder particles and abrasion of the walls of the drum by grinding bodies consisting of zirconium dioxide. The resulting mixture was subjected to uniaxial pressing to obtain blanks for further sintering [6]. The sintering process was carried out according to two schemes (table 3).

| Table 3. Physico-mechanical characteristics of ceramic samples |
|---------------------------------------------------------------|
| Sample       | Hᵥ₀,₃        | σᵥ, MPa  | KᵢC, MPa/m¹/₂ (bend) |
| Type I       | 958±99       | 122 ±13  | 4,0±0,37             |
| Type II      | 1336±144     | 139±11   | 4,5±0,21             |

Type I samples were prepared by uniaxial pressing of a mixture of powders followed by reaction sintering. Pressing was carried out in a mold under a pressure of 50 MPa. Sintering was carried out in a vacuum chamber for 30-120 minutes, at temperatures of 1700-1800 °C. In this case, the chamber was subjected to two successive cycles of "nitrogen purge" - pumping to a residual pressure of 10⁻²…10⁻³ mm. Hg. Art. The pressed blanks were placed in a crucible of boron nitride.

Type II samples were obtained by hot pressing of a mixture of powders at temperatures of 1700-1900 °C and a pressure of 500 kg/cm² for 12 minutes. A mixture of powders was placed in a graphite form, the walls of which were coated with an alcohol-based boron nitride coating to prevent the influence of carbon [6]. To eliminate the residual carbon introduced from the crucible, the samples were additionally annealed at 1900 °C. All samples of both types were subjected to grinding and polishing.

The obtained compacts and initial powders were studied using a Tescan Vega scanning microscope. The XRPA method was used to analyze the phase composition on a Bruker D8 ADVANCE diffractometer.

3. Results and discussion

The samples of the obtained ceramics have a gray color, densification during sintering did not occur completely, the physical and mechanical characteristics of the samples from our earlier works [6] indicate this. This is also clearly seen in the type I sample obtained by the reaction sintering method (figure. 2).
Figure 2. The imprint of the Vickers Indenter on a type I sample

Cracks didn’t originate in the corners of the imprint on this type I sample. This appears to be due to insufficient seal, and this method of measuring the fracture toughness is difficult to apply to ceramic materials with insufficient density (less than theoretical density, about 30-40%). The pattern in type II samples similar to what is presented in the literature (figure 3). Cracks, such as Palmqvist, extend from the corners of the imprint.

Figure 3. The imprint of the Vickers Indenter on a type II sample

To determine the crack resistance, formula (1) was chosen, since the crack is a Palmqvist type of crack [4, 7].

\[ K_{IC} = 0.018 \left( \frac{E}{H_v} \right)^{1/2} \frac{P}{c^2} \]

E- Young modulus, GPa ; \( H_v \) - hardness; \( P \) - force indenter, N; \( c \) - the distance from the top of the imprint to the end of the crack, mm.
The result is shown in (table 4). For comparison with the data obtained earlier, the bending test method was applied.

Table 4. Physico-mechanical characteristics of ceramic samples based on Al_{23}O_{27}N_{5}

| Sample | HV0,3 | σv, MPa | KIC, MPa/m^{1/2} (bend) | KIC, MPa/m^{1/2} (indentor) |
|--------|-------|---------|--------------------------|-----------------------------|
| Type I | 958±99| 122 ±13 | 4,0±0,37                | 3,52                        |
| Type II| 1336±144| 139±11 | 4,5±0,21                | -                           |

Taking the error into account, we can say that the crack resistance parameter measured by the indenter injection method repeated the result of crack resistance measured by bending a cracked sample. Accumulation of measurement statistics is required to confirm the efficiency of this method and to compare it with other methods for measuring crack resistance.

To assess the resulting crack resistance, the data obtained is compared with the results of other researchers in the field of mechanical testing of ceramic materials. For example, in the work of Gagotsi [1] there is a graph of the dependence of crack resistance on the length of the crack. For the data obtained we construct a point for the sample (red rhombus). This point falls on line 1b – this line is built for the hot-pressed Si_{3}N_{4}, similar in density and mechanical properties. This result indicates a certain degree of reliability of the result.

Figure 4. Comparison of literature data and the results obtained

4. Conclusions
The method of measuring crack resistance with the implantation of an indenter, followed by the calculation of crack resistance is a very convenient and fast method for monitoring ceramic samples.
The work compared the crack resistance obtained by two methods for two types of ceramics. For compacted ceramics, this method showed a result comparable to the results obtained in previous research, however, for samples with lower compaction, this method is unsuitable. The development of cracks directly depends on the compaction of the sample, since the pores are barriers to the propagation of cracks.

Many questions regarding the measured values of crack resistance remain unresolved at the moment: differences in the data obtained during the interaction of microcracks of various nature with obstacles, such as the grain boundary. The problem of a quantitative description of the effect of the load effect on indentation on the value $K_{1c}$ remains unresolved. The data obtained are questioned by indentation methods for materials in which the size of a unit of microstructure is close to the size of microcracks formed during indentation. With all this, indentation methods are very simple and convenient in practice and do not require the use of a large number and complex configuration of samples of the studied material, which makes methods for measuring crack resistance very promising.

This method for oxinitride and other structural ceramics may be preferable in cases where urgent determination of crack resistance and hardness as interrelated parameters is required. Literary data is required to be reproduced experimentally, in order to evaluate the results and determine the optimal technological regimes, the compositions of powder mixtures, the modes of synthesis, pressing and sintering. It should be noted that today there is no proven technology for the production of oxynitride aluminum ceramics, both in Russia and in other countries. Accordingly, research in the field of obtaining a transparent ceramic material from aluminum oxynitride is necessary, since this material can replace a whole class of transparent structural materials.

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