High-speed induction heating during tensile strength testing of a composite material based on an inorganic binder at temperatures up to 1500 °C

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Abstract. High-speed induction heating was used during strength tests in an oxidizing atmosphere of a composite material based on an aluminum-chromophosphate binder. Induction heating allows heating modes close to those of aircraft operating modes, which avoids the appearance of crystalline phases such as Al(PO₃)₃, Cr₂O₃, which arise under conditions of slow heating under classical methods of strength testing at elevated temperatures.

Keywords: alumochromophosphate binder, composite material, tensile strength, high temperature tests, induction heating

1. Introduction

The development of promising high-speed aircraft is associated with the problem of choosing structural materials that are resistant to high temperatures [1], in a wide range of heating rates, coupled with power loading. The solution to this problem necessitates the conduct of heat-strength tests of materials in order to determine the physical and mechanical characteristics. Classical methods of mechanical testing at elevated temperatures involve quasi-stationary heating. Under such heating conditions, the mechanical properties of the material may differ from the properties that are observed under actual operating conditions. Induction heating allows high-speed high-temperature heating of materials to high temperatures.

Induction heating is widely used as a method of heating conductive materials [2, 3]. The advantages of induction heating include low inertia and easy access to the sample, which facilitates the measurement. For non-conductive materials, induction heating can be used only in the presence of an intermediate heating conductive element heated by the inductor, from which the test sample is heated, [4, 5]. Heating through an intermediate heater leads to a certain increase in inertia (the smaller, the thinner the intermediate heater). The choice of material for the intermediate heater depends on the temperature and the medium in which the heating is carried out.

Composite materials based on inorganic binders are promising materials that can be used under conditions of high-temperature high-speed heating [6]. One of the representatives of this class of materials based on an aluminochromophosphate binder (ACPB) [7]. The study of the mechanical properties of this class of materials under high-speed high-temperature heating is an important task, since it allows one to obtain properties in conditions close to real operating conditions.
2. Materials and research methods

The object of the study was a composite material made by contact molding from an alumochromophosphate binder with the addition of Al2O3 powder, which is applied to quartz fabric (ACPB composite material).

The calculation of the temperature distribution in the sample during stationary and non-stationary heating was performed using ANSYS Fluent. The following characteristics were calculated:

1. - conditions for achieving a stationary heating mode, the required exposure time;
2. - temperature distribution in the sample.

Tensile tests were carried out on the tensile setup IR5047-50 at temperatures of 1100, 1200, 1300, 1500 °C. Loading speed 5 mm/min. 5 samples were tested for each temperature point. Temperature control was carried out with a Modline 5 partial radiation pyrometer. An HfB2 heater was placed between the grips (Figure 1) in thermal protection from porous Al2O3. The heating rate was 10 °C/s. Upon reaching the required temperature was carried out isothermal exposure for 180 seconds. After isothermal exposure, a tensile test was carried out. A schematic representation of a laboratory setup is shown in Figure 2.

![Fig. 1. HfB2 based heater](image1)

![Fig. 2. Schematic representation of a laboratory setup](image2)

Tensile strength σ, MPa is determined by the formula:

\[ \sigma = \frac{P}{S} \]

where P is the maximum load during tensile testing, N; S is the cross-sectional area of the working area of the sample, mm2.

X-ray phase analysis (XRD) was carried out on a PANalytical Empyrian Series 2 diffractometer in CuKα radiation with a nickel filter using the Data Collector, High Score Plus software package. To identify xrd patterns, the database “PDF2-2015” was used.

3. Thermophysical Calculation
The geometry and the designation of the elements are shown in Figure 3. The following characteristics were subject to a calculated assessment: (1) - conditions for achieving a stationary heating mode, the required exposure time (Figure 4); (2) - temperature distribution in the sample (Figure 5).

**Fig. 3.** The computational area and computational domains around the sample: 1 – air, 2 – thermal insulation, 3 – the sample, 4 – metallic uncooled grips, 5 – heater. The following notation is introduced for the boundaries of computational domains: B1 – thermal insulation-air boundary, B2 – axis of symmetry, B3 – the sample-air boundary, B4 – the grips-air, B5 – outer border of calculating domain for air, B6 – the heater – air, B7 – outer border of calculating domain for the grips.

The calculation showed that the exposure time of 180 seconds is sufficient to achieve uniform heating of the working area of the sample. It should also be noted that in the experiments, the heating rate was \( \sim 10 \, ^\circ\text{C} / \text{s} \), while in the calculation it was \( 67 \, ^\circ\text{C} / \text{s} \). Thus, the estimated time for reaching stationary heating is an upper bound. The calculation of the temperature distribution in the sample showed that the temperature difference across the layer thickness when entering the test mode does not exceed 10 degrees up to a sample surface temperature of \( \sim 1700 \, ^\circ\text{C} \).
4. Mechanical tensile tests
Table 1 presents the results of determining the tensile strength of ACPB composite material at various temperatures.

| Temperature (°C) | Average (MPa) | STD  | Coefficient of variation (%) |
|------------------|---------------|------|-------------------------------|
| 1100             | 15,0          | 1,5  | 10                            |
| 1200             | 14,3          | 0,7  | 5                             |
| 1300             | 13,4          | 1,2  | 9                             |
| 1400             | 7,5           | 0,8  | 10                            |
| 1500             | 8,1           | 1,9  | 23                            |

Figure 6 shows the samples after the test. The results are characterized by a low dispersion of strength values - up to 10% at test temperatures up to 1500 °C.

At a temperature of 1400 °C, a marked drop in strength occurs. This fact can be explained by the fact that at these temperatures, quartz fabric ceases to play a reinforcing role due to softening. In general, the nature of destruction changes (Figure 7). At a temperature of 1400 and 1500 °C, cracks are observed perpendicular to the length of the sample with a thickness of ≈ 300 μm. A change in the surface colour of the sample can be explained by a change in the valency of chromium, which is contained in the aluminochromophosphate binder.
5. X-ray phase analysis

The results of X-ray phase analysis of ACPB composite material after tensile tests are presented in Figure 8. According to the X-ray diffraction patterns, it is seen that after heating to a temperature of 1100 °C, the formation of the crystalline phase AlPO4 occurs. The shift in the position of the maximum of the main AlPO4 peak with increasing temperature is associated with the ordering of the structure (Figure 9). However, with high-speed induction heating, additional crystalline phases, such as Al(PO3)3, Cr2O3, do not appear, which arise under conditions of slow heating under classical methods of strength testing at elevated temperatures [7].
6. Conclusion
The developed prototype laboratory setup allows tensile tests to be carried out under conditions of high-temperature, high-speed heating in an air atmosphere to temperatures of about 1500 °C. During mechanical tests, it was revealed that at a temperature of 1400 °C, a change in the nature of failure occurs, which is accompanied by a decrease in strength by about a factor of two relative to tests at 1300 °C. This fact can be explained by the fact that at these temperatures, quartz fabric ceases to play a reinforcing role due to softening.
According to x-ray phase analysis, the appearance of crystalline phases in the area of induction heating is revealed. The crystalline AlPO_4 phase is already formed at a temperature of 1100 °C and its amount increases with increasing temperature. The shift in the position of the maximum of the main AlPO_4 peak with increasing temperature is related to the ordering of the structure.

With high-speed induction heating, additional crystalline phases, such as Al(PO_3)_3, Cr_2O_3, do not appear, which arise under conditions of quasi-stationary heating with classical methods of strength testing at elevated temperatures.

References

[1] Fahrenholtz W G and Hilmas G E 2017 *Ultra-high temperature ceramics: Materials for extreme environments*. Scripta Materialia, **129** 94–99. doi:10.1016/j.scriptamat.2016.10.018

[2] Hyde C J, Sun W and Leen S B 2010 Cyclic thermo-mechanical material modelling and testing of 316 stainless steel. International Journal of Pressure Vessels and Piping, **87-6** 365–372. doi:10.1016/j.ijpvp.2010.03.007

[3] Markovskya P E 011 Tailoring of microstructure and mechanical properties of Ti–6Al–4V with local rapid (induction) heat treatment // *Materials Science and Engineering A*, **2** 3079–3089.

[4] Neuman E W, Hilmas G E and Fahrenholtz W G 2016 Ultra-high temperature mechanical properties of a zirconium diboride–zirconium carbide ceramic. *Journal of the American Ceramic Society*, **99-2** 597-603.

[5] Yakushkin P Yu and other 2016 High-intensity induction heating as a way of approximating test conditions to operating conditions // New materials, S. 268-269 [in Russian]

[6] Chen N. et al. 2014 Studies on high-temperature thermal transformation and dielectric property of aluminum–chromium phosphates // *Journal of Thermal Analysis and Calorimetry*, **116-2** C. 875-879.

[7] Stepanov P A, Atroshchenko I G, Starodubtseva N I, Shutkina O V and Melnikov D A 2014 Development of high-temperature composite materials for heat-shielding and radio engineering purposes. // Promising materials, **10** S. 17-21 [in Russian]