Temperature dependences of strength properties and fracture mechanism of the Ni₃Al intermetallic compound synthesized under pressure

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Abstract. The article presents the results of a study of the strength properties and fracture mechanism of the Ni₃Al intermetallic compound samples synthesized under pressure at different test temperatures. Changes in the nature of the fracture mechanism in the test samples with an increase in the test temperature were revealed. It has been established that in the range from 20 to 1000 ºC there are two areas with different temperature dependences of strength properties.

1. Introduction

One of the traditional problems of modern materials science of high-temperature alloys is the task of increasing the strength of alloys operated under extreme operating conditions. One of the advanced solutions to this problem is the design of alloys with a low-dimensional grain structure. [1-2]. The above is especially true for nickel superalloys based on the Ni₃Al intermetallic compound, which are widely used in the space, aircraft, chemical and electronic industries, but which are extremely difficult to produce with low-size grain structures due to the low ductility of the intermetallic compound. [3-5]. The content of the intermetallic compound in modern superalloys can reach 0.89%; therefore, the application of known technologies for increasing the strength of alloys by forming low-dimensional grain structures by known methods of plastic deformation is not possible. [6-11].

The possibility of forming low-dimensional grain structures in intermetallic alloys is provided by the use of the technology of high-temperature synthesis of an intermetallic compound in a powder mixture of initial elements under pressure with high-temperature deformation of the synthesis product. [12-15]. The temperature-time conditions of the intermetallic alloy crystallization and the formation of its grain structure as a result of the exothermic reaction of the intermetallic compound formation determine the possibilities of severe plastic deformation of the high-temperature synthesis product and the formation of low-dimensional grain structures in the intermetallic alloy synthesized under pressure. By now, the temperature dependences of the strength properties of the Ni₃Al intermetallic compound obtained by vacuum melting and powder metallurgy are well known. At the same time, the strength properties of the intermetallic compound produced by the method of self-propagating high-temperature synthesis in a powder mixture of initial elements have been studied to a much lesser extent.

The aim of this work was to study the strength properties and the fracture mode of the Ni₃Al intermetallic compound synthesized under pressure in the temperature range 20 - 1000 ºC.
The paper is structured as follows: description of the method of high-temperature synthesis of Ni$_3$Al intermetallic compound samples in section 2; The results of the analyzes of samples obtained by the method of high-temperature synthesis under pressure were discussed in section 3; Section 4 presented the conclusions derived from the results of the study.

2. Materials and methods
High-temperature synthesis of the Ni$_3$Al intermetallic compound under pressure was carried out on experimental bench consist of hydraulic press equipped with a high-frequency generator for heating the steel die-mold prior to self-ignition of the compact from 3Ni + Al powder mixture (nickel particle size was 1 ÷ 3 µm, aluminum - 5 ÷ 10 µm). Additionally, the experimental stand equipped with a temperature recorder of heating the powder compact, a digital manometer for recording the pressure in the hydraulic system of the main cylinder of the press and the wake-up timer of the working pressure application in the main cylinder of the press (figure 1). The stoichiometric 3Ni+Al powder mixture was placed in a cylindrical steel die-mold with an internal diameter of 58 mm heated by high-frequency currents. Initiation of the expansion stroke of the hydraulic press and compacting of the high-temperature synthesis product occurred automatically according to the specified program of the technological cycle.

![Figure 1. Block diagram of an experimental stand for high-temperature synthesis of an intermetallic compound under pressure during continuous heating of the powder mixture of initial elements in a steel die-mold: working space of a hydraulic press, 2 — press master cylinder, 3 — steel die-mold, 4 — powder compact (a), scheme of intermetallic samples preparation for statistical evaluation of grain size. The arrow shows the sampling site for statistical evaluation of the grain size (b), scheme of a flat sample for tensile testing (c) and its photo (d).](image)

The grain structure of intermetallic samples was studied by optical metallography using ‘Neophot 32’ microscope on lamellar specimens cut from the central part of the synthesized compacts. Metallographic specimens were prepared by mechanical polishing with a gradual decrease in the size of the diamond abrasive particles of 1 micron. Grain structure was detected by argon ion etching at an accelerating voltage of 0.6 kV. Grain size determined by the method of random secants with averaging.
over 150 measurements. The phase composition of the samples was investigated by X-ray diffraction analysis using a ‘DRON-7’ X-ray diffractometer in CoKα radiation at an accelerating voltage of 40 kV and a current of 40 mA, the ‘Powder Cell 2.4’ software, and the ‘PDF 4’ database. The chemical analysis was performed using a ‘Carl Zeiss EVO’ scanning electron microscope equipped with an ‘Oxford instruments’ energy dispersive analyzer.

Strength and ductility of the synthesized product samples were evaluated on the basis of tensile curves. In order to obtain them, ‘dog-bone’ specimens had been prepared and tested using an ‘Instron 3360’ facility with a tensile rate of 0.2 mm/min at temperatures of 20 and 1000 °C.

Samples of the Ni3Al intermetallic compound synthesized under pressure and prepared for tensile tests were blades with holes for rigid fixation in grips.

3. Results and discussion

The results of X-ray diffraction study and analysis of the chemical composition (figure 2(a)) showed that the used synthesis procedure ensures complete transformation of the powder mixture into the Ni3Al intermetallic compound in the entire volume of the synthesized product.

![Figure 2](image_url)

**Figure 2.** The diffraction pattern (a) with the chemical composition and typical grain structure (b) of the Ni3Al intermetallic compound synthesized under pressure.

In figure 2(b) shows the characteristic grain structure of the Ni3Al samples synthesized under pressure. The grain structure of the samples is equiaxed, the range of grain sizes is 2–25 µm, and the average grain size is 6.82 µm.

The summary results of tensile experiments at temperatures from 20 to 1000 °C are presented on the histogram (figure 3), where σ0.2, σb, ε are the conventional yield strength, ultimate strength and elongation, respectively.

The values equal to one on the histograms correspond to the maximum indicators of strength properties - 550 and 570 MPa for the conditional yield point and ultimate strength at 600 °C and relative strain equal to 8% at 1000 °C. From the results presented, two characteristic stages of the change in properties from temperature are clearly noted. Up to 600 °C, the values of the yield stress and ultimate strength increase monotonically from 400 and 480 MPa to 540 and 570 MPa, respectively.

This effect is abnormal for the change in the deformation behavior of materials with increasing test temperatures. Simultaneously with an increase in ultimate strength indicators in this range, there is a slight decrease in the values of relative strain from 3.2 to 2.4%. In the range from 600 to 1000 °C, there is a sharp change in the temperature dependence towards a decrease in the yield stress from 540 to 115 MPa and the ultimate strength from 540 to 165 MPa with an increase in the values of relative strain from 2.4 to 8%.
Figure 3. Histograms of tensile strength properties of the Ni3Al intermetallic synthesized under pressure at temperatures of 20, 200, 400, 600, 800 and 1000 °C

In figure 4 shows profile (top row) and surface (bottom row) images of fractures of intermetallic specimens after stretching at temperatures of 20, 600, 800 and 1000 °C.

Figure 4. Optical images of surfaces of working parts, fractured specimens after tensile tests at different temperatures

It can be stated that the fracture of intermetallic specimens, regardless of the test temperature, occurs along grain boundaries. As the temperature rises, the proportion of intragranular fracture decreases with an increase in the proportion of brittle intergranular fracture. A decrease in the size of cracks and their number at the fracture surface of the samples means a change in the fracture mechanism with an increase in the test temperature, which changes from intragranular at temperatures up to 600 °C to intergranular at 1000 °C.

4. Conclusion
When tensile samples of the intermetallic compound at temperatures from 20 to 1000 °C, an anomaly in the deformation behavior was revealed in the form of a change in the fracture mechanism with increasing
temperature from intragranular (along grain boundaries) to intergranular (brittle fracture). Ni₃Al intermetallic synthesized under pressure has high values of strength properties.

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