Influence of the Thermal-force Effect on the Process of High-temperature Synthesis of the Ni_3Al Intermetallic Compound

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Abstract. In this work, the intermetallic compound Ni_3Al was obtained by high-temperature synthesis under pressure at various values of the preliminary pressure on the initial powder mixture (3Ni+Al). The study of pressure-time and displacement-time diagrams gave a coherent picture of the synthesis passage over time. It was found that an increase of preliminary pressure leads to a decreasing of the powder compacts porosity. In this regard, the smallest displacement of the press plunger after initiating the synthesis reaction in the powder compact was observed at the highest value of the preliminary pressure on the compact. The role of preliminary pressure on the initial powder mixture in the process of the grain structure formation of the Ni_3Al intermetallic compound synthesized under pressure was determined.

1. Introduction

The Ni_3Al intermetallic compound, related to alloys with high-symmetry L12 superstructure, is of considerable interest in modern materials science. Due to its outstanding properties such as high heat resistance, wear resistance, and anomalous temperature dependence of strength, Ni_3Al has gained widespread use as the basis for many modern high-temperature nickel superalloys used in aircraft, space industry, etc. [1-3]. At present, intermetallic compounds are obtained by various methods: electrolytic recovery of intermetallic crystals from melts, aluminothermic reduction of oxygen compounds, smelting in arc and induction furnaces, etc. Generally, traditional methods of synthesis, such as casting and melting, are not used in the manufacturing of most intermetallic compounds, mainly due to the large differences between the melting points and the densities of the initial components [4,5].

Currently, one of the most effective and versatile methods for producing Ni_3Al intermetallic compounds is the method of self-propagating high-temperature synthesis (SHS), based on the initiation of exothermic reactions of the chemical compounds formation in powder mixtures of the corresponding composition. The SHS method allows one to obtain not only various chemical compounds, including multicomponent compositions but also alloys and composite materials based on them [6]. To the advantages of the SHS method, its necessary include the high reaction rate of synthesis of chemical compounds (from several seconds to several minutes) and the relatively high purity of the final products [7].

The main directions in the development of production technologies and manufacturing of SHS products are theoretical and experimental studies of the kinetics of high-temperature synthesis of materials with a given structural and phase state and the use of SHS technologies in the materials science of products with unique physical and strength properties for extreme operating conditions. The prospects of this area of modern materials science are shown in fig.1 by data on the dynamics of the increase in the number of publications over the years over the past 10 years on the example of SHS known intermetallic compounds, SHS intermetallic compounds Ni_3Al and SHS as the transformation processes of the physicochemical state of disperse media under the conditions of the exothermic reaction to form the target chemical compound.

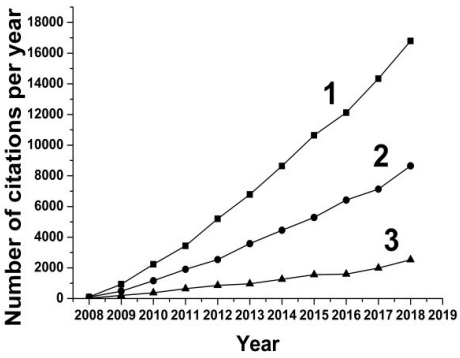

Figure 1. Dynamics of publication activity by year: 1 - SHS intermetallic compounds, 2- SHS intermetallic compounds Ni_3Al, 3 - SHS as a science about the processes of transformation of the physicochemical state of dispersed media under the conditions of the exothermic reaction of the target chemical compound formation (according to the Web of Science for February 2019).

A special place in the SHS technologies of the Ni_3Al intermetallic compound and alloys based on it is occupied by the SHS Ni_3Al technology under pressure, which allows the formation of billets or structural products. High speeds of phase transformations in the conditions of the bulk exothermic reaction of the intermetallic compound synthesis in the initial powder mixture create considerable difficulties in controlling the process of the structural-phase state formation and the strength properties of the synthesized under pressure intermetallic product. One of the ways to solve this problem is to use high-speed recording equipment of the volumetric exothermic reaction processes of intermetallic compound synthesis in the initial powder mixture, which allows registering temporal, temperature and power parameters of the processes of synthesis and compaction of the synthesis product in continuous shooting regime.

The purpose of this work is an experimental study of the influence of the thermal-force effect on the powder mixture of the initial elements on the parameters of the high-temperature synthesis process and the formation of a grain structure in a compact synthesized under pressure from the intermetallic compound Ni_3Al.

2. Material and methods

Samples of Ni_3Al intermetallic compound were synthesized in a powder mixture of nickel with aluminum of stoichiometric composition (nickel particle size of ~ 2.0 μm and aluminum ~ 1.0 μm) in cylindrical steel molds with an inner diameter of 58 mm on a hydraulic press by heating the molds by currents high-frequency till auto-ignition of powder compacting and force compacting of a high-temperature synthesis product. The process of the product's compacting was synchronized in time with the burning process of powder compact using an automatic control system of the preload on the initial powder compact, the heating speed of the powder compact and the digital recording system of the compaction front displacement of the thermo-sensitive mixture with a recording step of 0.25 s. The phase composition of the synthesized intermetallic compound samples was studied on an x-ray diffractometer DRON-7 in $Co_{K\alpha}$ radiation at an accelerating voltage of 35 kV and a current of 20 mA. The relative error in the measurements of displacements and pressures was no more than 2%. The grain size was determined by the method of random secants with averaging over 150 measurements.

3. Results and discussion

In fig. 2a is represented a diagram of a die mold for the high-temperature synthesis of intermetallic compounds under pressure. The performed diffractometric analysis of the obtained samples showed the identity of the phase composition of the samples synthesized at different preliminary pressures to the reference sample of stoichiometric composition (Fig. 2b).

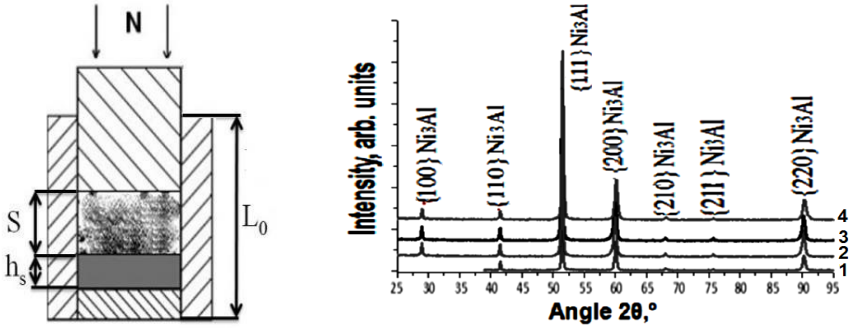

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Figure 2. The scheme of the die mold for high-temperature synthesis of intermetallic compounds under pressure (a), diffractograms from samples of intermetallic compound Ni3Al synthesized under pressure at various preliminary pressure values on the original powder compact: 115.1 MPa (1), 76.5 MPa (2), 32.6 MPa (3), Ni_3Al (reference) (4) (b).

At the first stage, studies of the synthesis passage in time were carried out for samples of the intermetallic compound Ni_3Al, depending on the value of the preliminary pressure on the initial powder compact. The high-temperature synthesis of the Ni_3Al intermetallic compound under pressure was implemented with the following sequence of steps of the synthesis process (Fig. 3 a, b):
(1) pre-pressure was applied to the powder mixture (3Ni + Al) in the die mold,
(2) self-ignition of the powder mixture was initiated by heating of the mold by high-frequency currents,
(3) upon ignition of the powder compact, the thermosetting powder mixture was compacted with a predetermined force of the hydraulic press.

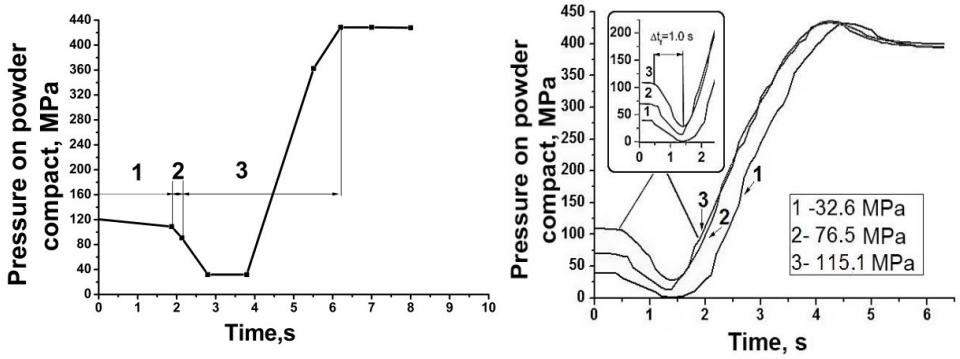

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Figure 3. Scheme of the high-temperature synthesis process of the Ni3Al intermetallic compound (a); the pressure-time diagrams of the high-temperature synthesis process of the Ni_3Al intermetallic compound with continuous heating of the powder mixture (3Ni + Al) and various values of the preliminary pressure on the initial powder compact (b), Δt_f – time to reduce pressure to a minimum.

The preliminary pressure on the powder mixture in the die mold ensures the formation of the optimum porosity of the powder compact in the range of 30-40%, in accordance with known conditions of the compact's ignition when heated to the melting point of the aluminum component [8]. From the moment when application synthesis product compacting pressure is applied to the thermo-reacting mixture, a high-precision recording of the hydraulic press plunger linear displacement occurs. In fig. 4 shows the time dependences of the plunger linear movement in the process of the synthesis product compaction at different values of the preload on the initial powder compact.

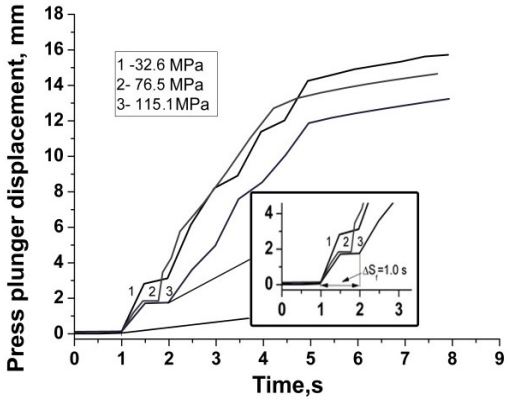

Figure 4. The time dependences of the hydraulic press plunger linear displacement in the process of the synthesis product compaction oat different preload values on the original powder compact: 32.6 MPa (1), 76.5 MPa (2), 115.1 MPa (3).

For shown in Fig. 4 dependencies are characterized by the presence of sites in time, due, apparently, with the formation of a liquid phase in powder compacts, which fills the pores of powder compacts. Similar sites were previously recorded on thermograms of high-temperature synthesis of Ni_3Al intermetallic [9]. Further, as the content of solid-liquid suspension containing intermetallic phases increases in the product of high-temperature synthesis, the process of the high-temperature synthesis product compaction accelerates up to the reaching the final value of the time dependence of the press plunger linear displacement during the formation of an intermetallic compound of a given composition in the entire volume of the synthesized compact. The difference in the size of the sites is due to the difference in the porosity values of the powder compacts - the smallest displacement of the press plunger after initiating the synthesis reaction in the powder compacting occurs at the highest value of the preliminary pressure on the compact at 115.1 MPa, which is caused by the highest compacting density. This regularity determines the nature of the time dependence of the relative compaction of the powder mixture and the dependence of the maximum relative compaction of the powder compact on the value of the preliminary pressure, which was calculated by the formula

$$\delta_i = \frac{S_i}{S+h_s}$$

where δ_i - is the relative compaction of the powder mixture at the current time, S_i - is the compaction of the powder mixture at the current time in the synthesis process, S - is the final compaction of the powder mixture during the synthesis, h_s - is the thickness of the obtained sample of the intermetallic synthesis product.

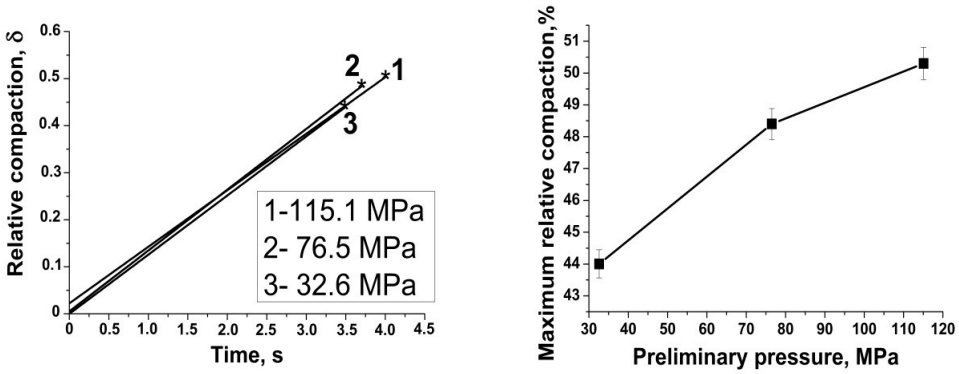

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Figure 5. The time dependence of the relative compaction of a powder compact during the high-temperature synthesis of the Ni_3Al intermetallic compound: 115.1 MPa (1), 76.5 MPa (2), 32.6 MPa (3) (a) and the dependence of the maximum relative compaction of the powder compact on the pre-pressure (b).

Based on the data on the time dependencies of the relative compaction at the current time (Fig. 5a), the dependence of the maximum compaction of the original powder compact on the value of the preliminary pressure (Fig. 5b) is constructed, from which it follows that the maximum compaction corresponds to the maximum preload on the compact. This is more clearly seen in the behavior of the speed and acceleration

curves of powder compacting: the highest value of the speed of synthesis product compaction corresponds to the minimum amount of preliminary pressure on the powder compact. This is due to the longer duration of the high-temperature synthesis process of an intermetallic compound under pressure at the maximum porosity value of the powder compact (Fig. 6 a, b).

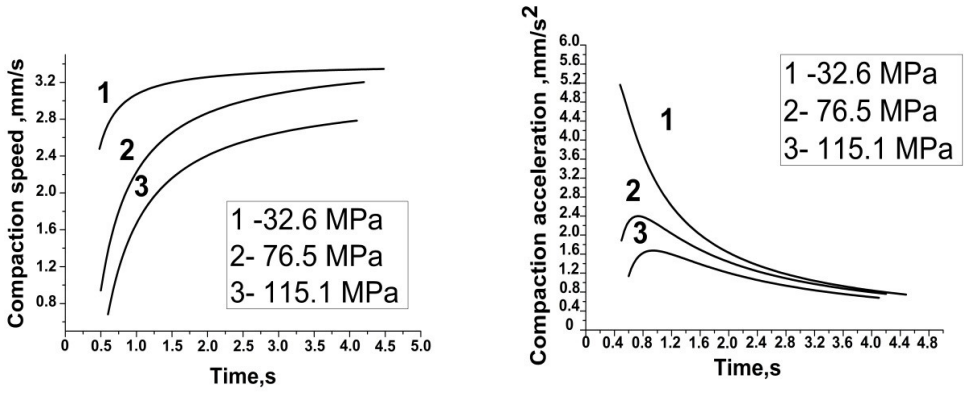

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Figure 6. The time dependences of the compaction speed of the powder compact (a) and the compaction acceleration of the compact (b) at different values of the preliminary pressure on the compact.

In other words, in the case of initiation of high-temperature synthesis in a powder compact with the highest porosity, the thermo-reactive powder mixture exists at high temperatures for the longest time, which determines more favorable conditions for grain growth in the intermetallic compound synthesized under pressure. Indeed, as shown in [10], a decrease in the preload on the initial powder compact leads to an increase in the grain size in the intermetallic compound synthesized under pressure. For samples synthesized with a preliminary pressure value of 115.1 MPa, the average grain size was the smallest, and the value of the intermetallic compound ultimate strength was greatest (table 1).

Table 1. The values of the average grain size and ultimate strength for Ni_3Al samples synthesized at different values of preliminary pressure [7].

Preliminary pressure, MPa	32.6	76.5	115.1
Average grain size, μm	10.5 ±0.7	8.7 ±0.5	3.8 ±0.4
Ultimate strength, MPa	336±6	406.3±8	482.35±9

4. Conclusion

The kinetics of the high-temperature synthesis process of the Ni_3Al intermetallic compound in a powder mixture (3Ni + Al) under pressure under the conditions of a volumetric exothermic reaction of the intermetallic compound formation is largely determined by the values of the preload on the original powder compact and the relative compaction. A comprehensive study of the process of shrinking the initial powder pressing throughout the process of an intermetallic compound high-temperature synthesis under pressure makes it possible to identify and evaluate the role of preload on the initial powder mixture and compaction rate in the formation of the grain structure and strength of the intermetallic compound Ni_3Al synthesized under pressure.

Acknowledgments

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