Effects of Inert Nanoparticles of High-Melting-Point Compositions on Grain Structure and Strength of Ni3Al Intermetallic Compounds

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Abstract. The paper represents experimental findings both in the area of effects of nanoparticles of inert high-melting-point TiN compounds on a Ni3Al intermetallic grain structure creation in the conditions of high temperature synthesis under pressure, and in the area of impact of grain structure modification on intermetallic compounds' strength factor temperature dependence. It was demonstrated that appending a stoichiometric composition of nanosized particles of high-melting-point inert chemical compounds (TiN) initiates a many-fold loss of average size of grain of Ni3Al intermetallic compounds, synthesized under pressure, as well as a sufficient intermetallic compounds' strength rise within a wide range of temperatures (up to 1 000 degree C). Electron-microscopic evaluations of a synthesized intermetallic structure with TiN nanoparticles, showed that, during the process of intermetallic polycrystalline structure creation from high temperature synthesis products melts, TiN nanoparticles are mainly spread throughout the boundaries and joints of grain structure, acting as stoppers of grain boundaries migration.

Introduction

Physical and mechanical properties of nickel alloys, which are widely used in engineering, substantially depend on their grain structure dispersability and morphology [1, 2]. Grain structure dispersability growth causes a sufficient rise of flow and strength limits, and nickel alloys ductility advance. Grain refining is one of the most efficient ways to enhance durability in the conditions of cyclic stress [3, 4].

It is possible to purposefully increase nickel alloys grain structure dispersability via nucleation and grain growth processes control, when alloys are being crystallized by adding highly dispersive particles of infusible chemical compounds, which are important parts of crystallization centers and efficient growth barriers of grains spread throughout the boundaries [5].

Above mentioned is of burning importance for heat-resisting alloys based on Ni3Al intermetallic compound, the amount of which can reach 89% in alloys. Extremely low ductility of intermetallic
compounds causes their tendency for brittle intercrystalline failure and loss of alloys strength in the conditions of high temperatures [6, 7]. Intermetallic alloys mechanical properties enhancement technologies focus mainly at controlled alloying and crystallization of alloys; ways of producing monocrystal articles; and at reinforcement of alloys with refractory particles or fibers [8]. At that a possibility to increase intermetallic alloys' strength by means of a intermetallic compound (the main alloy ingredient) grain structure atomization has been left unattended. Currently the world practice does not possess any technologies that allow enhancing intermetallic compounds mechanical properties via their grain structure atomization.

The paper presents research findings that demonstrate the way nitride TiN refractory chemical compound's nanosized particles (as potential centers of intermetallic compound crystallization and definite grains growth stoppers within the process of a intermetallic compound grain structure development, when it is being crystallized from melt) influence a grain structure dimension, as well as Ni3Al intermetallic compounds physical and mechanical properties [9].

Study Materials and Methods of the Experimental Research

Ni3Al intermetallic compound patterns were obtained through the method of high temperature synthesis in a nickel (~2.0 µm) powder mixture with stoichiometric aluminum (~1.0 µm) in the conditions when steel mould was subjected to a continuous heating with high-frequency current up to the moment when a 30% porosity powder compact autoignites with further pressure to the high temperature synthesis article [10, 11, 12]. Nanosized particles of a titanium nitride TiN (Figure 1) in a volume of 0.1 mas. % were added into an initial powder mixture of nickel and aluminum within the phase of mixing powders. To reach a distribution factor of TiN nanosized particles in a mixture of nickel and aluminum powders of a micron range of sizes, the mixture was carried out through two periods: by means of a vibromixer in a tank with a powder mass dissector, blending them in a multi knife homogenizer when they were in a dispersed dry-cleaning liquid (acetone) condition of a powder mixture. X-ray phase analysis of synthesized patterns of an intermetallic compound was carried out in the conditions of Cu Kα rays at 40 kV and 40 mA. Intermetallic alloys grain structure was investigated through the methods of optical metallography and scanning electron microscopy after metallographic specimen had been subjected to ion beam etching. Intermetallic compound patterns ware-resisting properties were tested with a BK -10 hard alloy counter body that moved along cutting tracks through the surface. The BK -10 hard alloy counterbody was a 3 mm ball that moved at 8N load with a linear speed equal to 2.5 cm per second with a 2.5 mm radius of a cutting ring, and with the total number of rotations that equaled to 3 500 revolutions. The intermetallic compound patterns stretch strength was evaluated at a 0.2 mm per minute rate of stretch.

Figure 1. TiN nanoparticles overview (transmission electron microscopy).
Results and Discussion

Figure 2 introduces diffractograms obtained from the Ni3Al intermetallic compound reference specimen and from the intermetallic compound patterns synthesized under pressure in a powder mixture of nickel and aluminum with 0.1 % of nanosized particles of a TiN titanium nitride adding.

![Diffractograms](image)

**Figure 2.** Diffractograms obtained from the reference specimen (1), from the patterns synthesized under pressure (2), and from the patterns synthesized under pressure with a 0.1 % of nanosized particles of a titanium nitride adding (3) of Ni3Al intermetallic compound.

The analysis of diffractograms introduced in Figure 2 allows making a conclusion that Ni3Al intermetallic compound patterns synthesized under pressure, including those which contain titanium nitride nano particles, have a one-phase composition.

Metallographic evaluation of intermetallic compound patterns’ grain structures showed that adding 0.1% of nanosized particles of a titanium nitride into the initial powder mixture of nickel and aluminum, we sufficiently influence the size of an intermetallic compound grain structure, i.e. the average size of a grain reduces from 10 µm to 2 µm (Figure 3).

![Metallography](image)

**Figure 3.** Metallography of a synthesized under pressure Ni3Al intermetallic compound grain structure (a) and a synthesized under pressure intermetallic compound which contains 0.1% of TiN nanoparticles (b), scanning surface electron microscopy of a synthesized under pressure Ni3Al intermetallic compound+0.1% of TiN nanoparticles (c).
Evaluations of intermetallic compound patterns (scanning electron microscopy) fracture relief thin structure showed that there are nanosized inclusions throughout the boundaries of intermetallic compound grains. An element compound energy-dispersive analysis technique helped to determine that nanosized inclusions consist of titanium and nitrogen, and are the nanoparticles of a TiN titanium nitride. Figure 4 represents micro photographs of a fracture surface of an intermetallic compound with TiN nanoparticles adding. Figure 4 b, d shows an energy spectrum of a grain body element compound and TiN particles at the grain boundary.

![Figure 4](image)

**Figure 4.** Fracture surface of a synthesized under pressure Ni3Al intermetallic compound with TiN nanoparticles (marked with arrows) (a, c) and energy spectra of a grain body element compound (b), and titanium nitride particles at the grain boundary (d).

TiN particles distribution throughout the boundaries and joints of grains in a synthesized Ni3Al intermetallic compound might be proved by the results of the investigations of intermetallic compound patterns' thin structure through the method of a transmission electron microscopy. Figure 5 presents both an electron-microscopic image of a TiN particle, situated within the joints of several grains of an intermetallic compound, and a particle element analyses findings.

Presented modification of a grain structure of a Ni3Al intermetallic compound, synthesized in a powder mixture of nickel, aluminum and TiN nanoparticles sufficiently effects intermetallic compound strength properties. Figure 6 represents temperature dependence of strength of a Ni3Al intermetallic compound, synthesized under pressure.

Strength temperature dependences of synthesized under pressure Ni3Al and Ni3Al+0.1%TiN intermetallic compounds show that an intermetallic compound grain structure modification with titanium nitride nanoparticles causes a sufficient (two times and more) increase of an intermetallic compound strength within wide range of test temperatures, which exceeds existing strength factor of
nickel aluminide patterns, obtained through the latest technologies [12]. At that when the temperature comes at 1000 degrees C the effect of grain structure modification on an intermetallic compound strength stops completely, which reveals differences in fracture mechanisms of the investigated intermetallic compounds at temperatures below 1000 degrees C.

Figure 5. Electron-microscopic image of a TiN particle at the joint of several Ni3Al intermetallic compound grains; and an energy spectrum of a particle element compound.

Fig.6. Temperature dependences of a Ni3Al intermetallic compound strength (1) and a Ni3Al intermetallic compound with 0.1% of TiN nanoparticles adding (2).

Conclusion
1. Appending a stoichiometric composition of nanosized particles of a titanium nitride into a powder mixture of nickel and aluminum causes a many-fold loss of an average size of a grain in Ni3Al intermetallic compound, synthesized under pressure.

2. A modificative effect of TiN nanoparticles on a Ni3Al intermetallic compound grain structure is determined by the character of their dissemination in a TiN particle, which appear during the process of intermetallic compound crystallization from melt. TiN nanoparticles are mainly placed along the
boundaries and in grain structures joints, preventing grain growth when a polycrystalline structure of an intermetallic compound is being developed.

3. A many-fold loss of an average size of a grain in Ni3Al intermetallic compound, synthesized under pressure, with TiN nanoparticles adding into the initial powder mixture of nickel and aluminum increases an intermetallic compound strength within a wide range of temperatures (up to 1 000 degree C) twofold and more.

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