

Experimental and Theoretical Study of Mass Transport during Annealing of Mechanically Activated Composite Granules of Ni–Al System

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Abstract— *The article discusses the process of internal energy accumulation during nickel-aluminum composite granules mechanical activation and the mechanism of primary energy accumulation associated with nickel nanoparticles formation in aluminum by the diffusionless mass transfer. The low level of activation energy of the diffusion process of nanoparticles dissolution with formation of an unsaturated solid solution provides energy release in the form of composite granules spontaneous self warming during the annealing at the temperature of 150°C. The cycle of mechanical activation - low-temperature annealing can be repeated several times, until the saturated solid solution of nickel in aluminum formation. After that, nickel nanoparticles diffusionless implicated in saturated solid solution will not dissolve in it during low-temperature annealing. The mathematical model of the processes has been developed. The estimation of the energy characteristics of the identified phenomena was done.*

KEY WORDS: Composite granules, Diffusionless process, Diffusion mass transfer, Energy storage, Mechanical activation, Relaxation.

I. INTRODUCTION

High-performance, cost-effectiveness and quality of the products in combination with the technological flexibility of production distinguishes and defines good development prospects of mechanochemical synthesis of a wide range of compounds in any scale of production - from grams to tens of tons of product. Full cycle of mechanochemical synthesis includes starting powders high-energy mechanical treatment (mechanical activation) until the desired compounds formation directly in the working area of the activator. Mechanical activation (MA) can be stopped at an earlier stages in order to unite the processes of monolitization and synthesis and realize other methods such as SHS. In any case, the main factor of the process efficiency - the maximization of processed materials reactivity, is determined by the power of a chemical reaction and the degree of disequilibrium progress state.

II. MECHANICAL ACTIVATION AND SUBSEQUENT ANNEALING

The reactive components mechanical activation will not change the total amount of the chemical interaction potential

energy, which is defined by heat output of the reaction, the nature, weight and the ratio of reactants. However, it can significantly accelerate the processes of interaction between components, reduce energy loss and improve the overall efficiency of chemical reactions by increasing the area of mutual contact of the components forming the "clean" surface contact, reducing the thickness of the interacting layers, a sharp decrease in the heat loss. Furthermore, there is an accumulation of additional internal energy in the mechanical activation process due to the formation of crystals of non-equilibrium concentration of activated linear and point defects (dislocations, ionic and atomic vacancies, interstitial atoms and ions ragged interatomic bonds). Energy is accumulated in the formation of structures in shear crystals changing angles between the atomic chemical bonds, and also due to the rotational defects, etc.

The vast variety of energy accumulators corresponds to a vast variety of conditions of their release. Extremely high degree of plastic deformation achieved during the mechanical activation transits metal in a highly thermodynamically unstable state. The internal stress and defects relaxation requires different activation energy and, therefore, occurs in various temperature ranges up to a temperature of recrystallization. With increasing annealing temperature and achieving the required speed of heat treatment, there occurs a consistent relaxation of additional internal energy from energy accumulators with self-heating of the mechanically activated materials and the dissipation of heat into the environment. [1–9].

In previously conducted work [10] it was shown that after 20 minutes of treatment, the initial powders form composite granules (CG). The degree of CG mechanical activation can be estimated by the front propagation velocity (FPV) in one-dimensional organized process of energy release. In the Ni - Al system, the FPV of nickel monoaluminide synthesis continuously increases with increasing MA time, reaches its maximum and drops to zero due to the occurrence of chemical interaction in the drum of a planetary mill.

The intermetallic compounds of different stoichiometry (Al₃Ni, NiAl, Ni₃Al) formation is defined by blending the appropriate ratio of the starting components, and have a different character of changes in activity, indicating that there are significant differences in their energy storage and energy

release during the MA (Fig. 1).

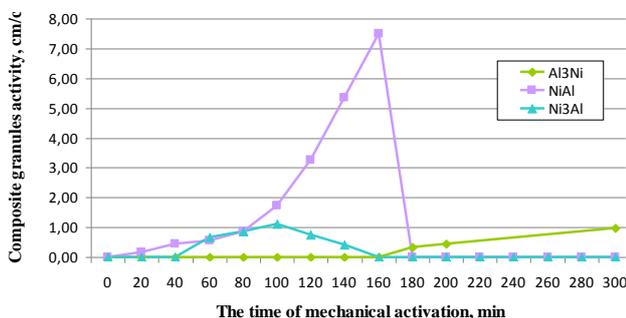


Fig. 1. Typical curves of composite granules activity changes during mechanical activation of Ni - Al system samples [10].

CG activity was found to have decreased depending on the temperature and annealing time, and their rate of heating. Energy release during annealing is shown in the form of self-heating of the composite granules and overheating about relative annealing temperature. Depending on the rate of heating oven superheat level varies from a few to several hundred degrees. At low heating rates, self-heating is almost completely compensated by the heat sink to the environment. At high heating rates, the heat sink does not manifest itself, and there is a rapid self-heating of the composite granules to a temperature overheating (1000 - 1500) °C. At an average speed of heating self-heating temperature reaches CG overheating at (100 - 150) °C. Thus composite granules annealing reduces their activity releasing energy from mechanical activation in the environment without changing the potential energy of chemical interaction at low and medium heating rates, and with the initiation of chemical reaction synthesis at high heating rates. These results do not explain the mechanisms for implementing the identified patterns, but define a picture how mechanical activation carries diffusionless mass transfer and forms by mechanical mixing of the starting components composite granules with a layered structure (Fig. 2), which has a high degree of disequilibrium and energy saturation [11].

In [12] the structure of Ni - Al mechanically activated granules revealed the presence of smoke-like clusters of nanoparticles (absent in the original mixture), which the authors attributed to some intermediate Ni-Al intermetallics or Ni solid solution in Al. Authors studied the sequence of formation of intermetallic compounds of different stoichiometry and temperature ranges shown their allocation. But the transition of nickel nanoparticles to solid solution has not been fixed.

Subsequent annealing of mechanically activated CG implement the mechanism of diffusion mass transfer, which return the system to a state of equilibrium. Depending on the temperature and processing time, a relaxation of the energy accumulators lead to self-heating of the composite granules and the dissipation of heat into the environment. The questions of specific mechanisms of accumulation - allocation of internal energy, "capacity" of the respective accumulators

and a possible longer time of powders mechanical activating to achieve a higher level of thinning layers in the CG and increase their energy level remain open.

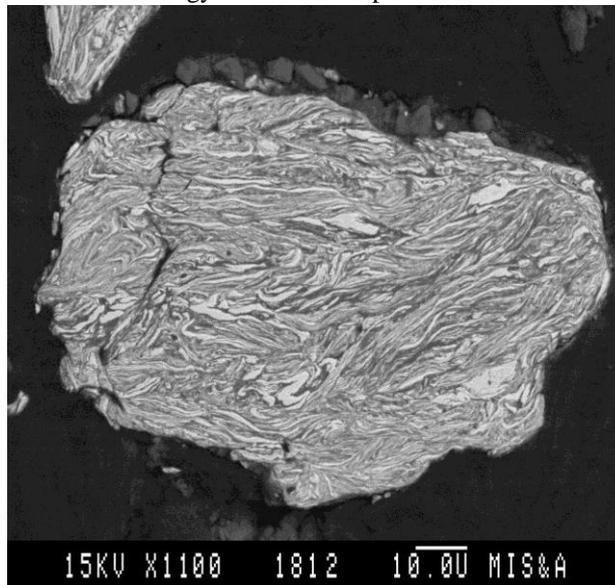


Fig. 2. Typical microstructure of the Ni - Al composite granules [11].

In this paper we propose a modified cyclic mechanical activation procedure for identifying the mechanism of formation during the mechanical activation and relaxation in low-temperature annealing of low-temperature phenomena of accumulation of additional internal energy.

III. METHOD OF THE LIFE CYCLE DETERMINING OF THE LOW TEMPERATURE ACCUMULATOR OF INTERNAL ENERGY

We suggest that diffusion less mass transfer during the mechanical activation process creates a two-tier structure consisting of alternating layers of nickel and aluminum of a few micrometers thickness, between which, in the aluminum interlayer the smoke-like nanostructures of the nickel particles are presented. Nickel nanoparticles, surrounded by aluminum are the most active layer with a nonequilibrium structure.

Experiments have shown that annealing at a temperature of 150 °C is sufficient to activate the process of dissolution of nickel nanoparticles to form a solid solution of nickel in aluminum. After annealing, the source of self-heating disappears, and the same granules can again be subjected to mechanical activation in order to form a finer structure of alternating layers without risk of the initiation of chemical interaction in the working volume of the activator. It was necessary to determine experimentally the energy parameters of this transition and its implementation mechanisms.

Studies were conducted on composite granules formed during mechanical activation of the initial mixtures to obtain a final compound NiAl from powders of Ni and Al, the characteristics of which are presented in Table 1.

Table 1 Properties of the starting powders of the particles and compositions of the samples burdening

Properties	Al	Ni
Material grade	АСД-1	ПНК-УТ3
Specifications	ТУ 5494-95	ГОСТ 9722-97
Mass fraction, %	Not less than 99,7	Not less than 99,9
Particle size, μ	15 – 50	5 - 15

To carry out the MA and produce composite granules we have used planetary mill Retsch PM 400. As grinding bodies there were steel balls with a diameter of 10 mm. Activation mode was selected with filling not more than 20% of the activator, the ratio of "grinding bodies: the activatable material" - 20: 1, the weight of powders – 35 g, processing environment - argon. Evaluation activity of CG was estimated by the velocity of the reaction front of NiAl compound synthesis propagation.

Structural studies were performed using optical and scanning electron microscopy. X-ray diffraction analysis was performed on a diffractometer ADVANS BRUKER D8 using a monochromator on the diffracted beam and the characteristic radiation (wavelength 0.154178 nm) in step mode and continuous writing.

The experimental technique includes the initial powders and composite granules mechanical activation to achieve a high degree of activity without initiating a chemical reaction with subsequent annealing at different temperatures. Next, the whole cycle was repeated several times. Experimental results are presented in Table 2 and Fig. 3.

Table 2. Data for assessing the Ni-Al composite granules activity

Annealing temperature, °C	Composite granules activity, cm/c					
	First cycle		Second cycle		Third cycle	
	Activation 1	Annealing 1	Activation 2	Annealing 2	Activation 3	Annealing 3
150	6,3	4,7	5,8	4,5	6,25	2,66
200	7,1	1,8	4,1	—	—	—
250	7	0,7	0,8	—	—	—

The overall picture of the transition of diffusionless to diffusion mass transfer is as follows: the starting powders mechanical activation during the diffusionless mass transfer leads to the formation of composite granules with a two-tier structure and high level of activity (the non-equilibrium state of the system). At low temperature (150 - 180) °C annealing of active composite granules the process of diffusion mass transfer is implemented, wherein smoke-like nickel nanoparticles reacts with aluminum and form an unsaturated solid solution of nickel in aluminum (equilibrium state of the system). After repeated mechanical activation of annealed composite granules, they again become very active, indicating the formation in the Al part of layered Ni-Al structure the new portion of smoke-like nickel. Repeated low-temperature

annealing is not much different from the primary except the interaction of nickel nanoparticles not with pure aluminum, but with the unsaturated solid solution of nickel in aluminum, formed at the previous stage. This cycle of mechanical activation - low-temperature annealing can be repeated several times, until the formation of the saturated solid solution of nickel in aluminum. After that, nickel nanoparticles diffusionless implicated in saturated solid solution will not dissolve in it during low-temperature annealing.

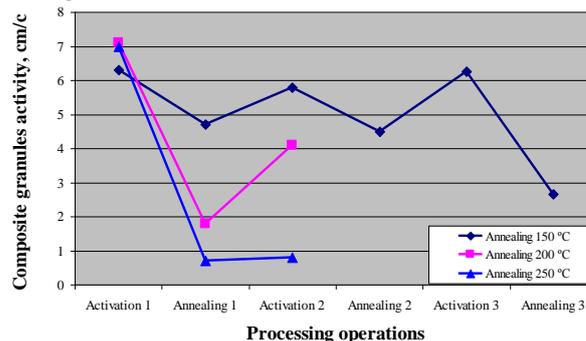


Fig. 3. The composite granules activity achieved after processing operations at various annealing temperatures.

Given these data we can build a sequence of events leading to the initiation of chemical interaction in the working volume of the planetary mill. Mechanical activation is a flow of energy, aimed at the deformation of composite granules. A very high degree of plastic deformation of CG at relatively low temperatures in the process of forming granules of non-equilibrium two-tier structure of alternating layers of the starting components with additional kneading nickel nanoparticles into the aluminum. During processing, due to the fluctuations of the energy flux in the micro regions of nanosmoke reached the activation energy of diffusion transition in solid solution and part of the nickel nanoparticles continuously dissolves, releasing energy, which leads to local overheating. If the temperature of local overheating is insufficient to initiate the dissolution of neighboring particles, the process will stop. As machining nanosmoke density increases. Increasing the content of nickel in aluminum in the clouds of nanosmoke increases the likelihood of initiating a process of diffusion of dissolution of neighboring particles. The process assumes the character of a chain reaction and at a certain point in time when the rate of the branching chain reaction exceeds the critical value, the entire volume of the cloud nickel nanodoma almost instantaneously dissolved in aluminum. Providing that energy will be added to the total energy of mechanical activation and self-heating reaches the level of the activation energy of a chemical reaction synthesis of intermetallic corresponding temperature. Next, again a chain reaction, but at the level of the layers in close contact of the starting components. Released during the synthesis of intermetallic energy leads to overheating, the corresponding activation energy of the synthesis of the following intermetallic. Avalanche process evolves and comes to the

formation of the final monoaluminide NiAl nickel directly in the working volume of mechanical activation of installation.

This development allows us to recommend a new technological cycle of mechanical activation in the processing of nickel - aluminum. The process must be interrupted when the activity close to critical. Composite granules should be subjected to low temperature annealing. Cycles mechanoactivation - annealing will increase the overall processing time of composite granules to achieve the formation of a thin layer of reactive components and accumulate, eventually more inner energy to the follow-furnace processes of forming a monolithic material.

IV. METASTABILITY EFFECTS SIMULATION. THE PROCESSES OF NICKEL - ALUMINUM INTERMETALLIC SYNTHESIS

We assume that after the mechanical activation process conducted with temperature control, the initial powder mixture with a predetermined volume fraction of nickel goes into system of composite granules with the layered structure which is shown in Figure 2. The density of typical boundaries depends on the time of the activation process, the volume fraction of the nickel, activation temperature, and so forth.

Let's consider the typical conditional boundary in the composite granule formed after mechanical activation process for the system nickel - aluminum. Recently in the work [12] it was reported that in the vicinity of each boundary in a layered system of granules the special nanostructure from the nickel nanoparticles are formed. Such nanosystem can be considered as a nanocomposite with the aluminum matrix "reinforced" by the nickel nanoparticles. The strain energy of such nanocomposite is the sum of the energy of deformation of aluminum matrix, nickel particles and surface energy associated with interfaces Al - Ni. The considered nanosystem is the metastable and the transition through some energy barrier goes into solid solution of Ni in Al. In this work we show that after process of transition into solid solution the some energy is released. This energy naturally associates mainly with the surface energy, because in a solid solution of nickel atoms partially replace aluminum atoms without forming an additional phase, which can be considered as the inclusion.

We assume that the structure of the typical granule (see Fig.2) is determined by the specific density of interfacial boundaries: F/V , F/V is the volume of the granules, the F the surface of the contact boundaries between phases. We introduce a parameter $r = V/F$ which is is the reciprocal to the specific density of the boundaries. During mechanical activation the specific density of boundaries grows, therefore, the parameter $r = V/F$ decreases with increasing duration of activation. Finally, we introduce a dimensionless parameter $d = r/l$, where l is the scale parameter of the nanocomposite Ni - Al which is formed in the vicinity of the phase boundary. We assume that the nanocomposite structure changes slightly in the process of mechanical activation and,

consequently, $l = \text{constant}$.

In the works [13-15] the variational model deformation of the materials have been elaborated, taking into account the scale effects and surface adhesion effects in heterogeneous materials with a developed structure. The dimensional analysis given in these works, allow to write the following expression for the potential energy:

$$U = U_v^0 + U_f^1 l + (U_v^2 + U_f^2) l^2 + U_f^3 l^3 = U_v^0 [1 + a_1 d^{-1} + a_2 d^{-2} + a_3 d^{-3}], \quad (1)$$

$$a_1 = \frac{U_f^1 V}{U_v^0 F} = \frac{(U_f^1 / F)}{(U_v^0 / V)}, \quad a_2 = \frac{(U_v^2 + U_f^2) V^2}{(U_v^0 + U_f^0) F^2} = \frac{(U_v^2 r^2 / V)}{(U_v^0 / V)} + \frac{(U_f^2 r / F)}{(U_v^0 / V)},$$

$$a_3 = \frac{U_f^3 V^3}{U_v^0 F^3} = \frac{(U_f^3 r^2 / F)}{(U_v^0 / V)}$$

Here U_v^0 is the potential deformation energy defined by the classical theory of elasticity, U_v^1 is the potential energy of the gradient effects defined by the cohesion scale effects in the volume of the body, U_f^1 is the interfacial potential energy (ideal adhesion [13]), U_f^3 interfacial energy defined by the gradient effects on the interphases and U_f^2 is the part of adhesion energy associated with interaction of ideal and gradient adhesion on the surface.

It was proved [15], that values U_v^0 and a_1 are always the positive values. At the same time, a_2 and a_3 the parameters that can be either positive or negative. Consider the typical dependences that show a change in the normalized energy U/U_v^0 of the dimensionless argument $d = r/l$. Parameter $d = r/l$ is a characteristic of the process of mechanical activation and decreases with increasing duration of this process. The curves of the fig. 4 show that the one-parameter ($a_1 = 1, a_2 = a_3 = 0$), a two-parameter ($a_1 = 1, a_2 = 1, a_3 = 0$) and a three-parameter representation of the potential energy ($a_1 = 1, a_2 = 1, a_3 = 1$) allow to simulate the growth of the potential energy with increasing duration of mechanical activation process.

As a result, we can consider equation (1) as a one-parameter, two-parameter and three-parameter models of deformation of composite granules during mechanical activation. Dimensionless parameters a_i generally determine the state of composite granules for the each activation time and can be estimated by the experimental dates.

Assume that the initial process is characterized by the parameters $U_v^{0(1)}, a_1^{(1)}, a_2^{(1)}, a_3^{(1)}$ and l . This process is shown by the dotted line on the Fig. 5. Upon annealing, which initiates the diffusion of nickel in aluminum, the other composite system is obtained, which is described by the other parameters $U_v^{0(2)}, a_1^{(2)}, a_2^{(2)}, a_3^{(2)}$ and l (solid line in Fig. 5). Hence, the dependence $\bar{U}(d)$, ($\bar{U} = U/U_v^0$) will have a discontinuity on the value $\Delta U = U^{(1)}(d_*) - U(d_*)$ for some parameter of the process $d = d_*$.

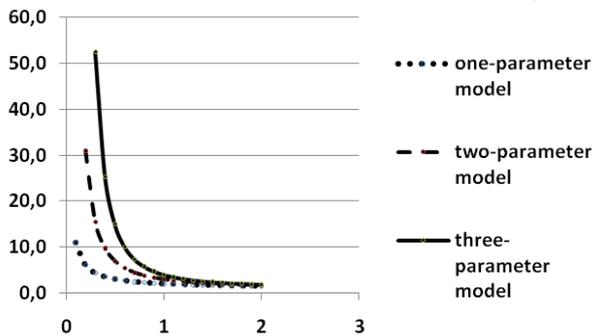


Fig.4. Potential energy dependences from parameter $d = r/l$ (duration of mechanical activation process); dotted line corresponds to the one-parameter model, ($a_1=1, a_2=a_3=0$), dashed line corresponds to the two-parameter model of changing of potential energy during the mechanical activation process, ($a_1=1, a_2=1, a_3=0$) solid line corresponds to the three-parameter model ($a_1=1, a_2=1, a_3=1$)

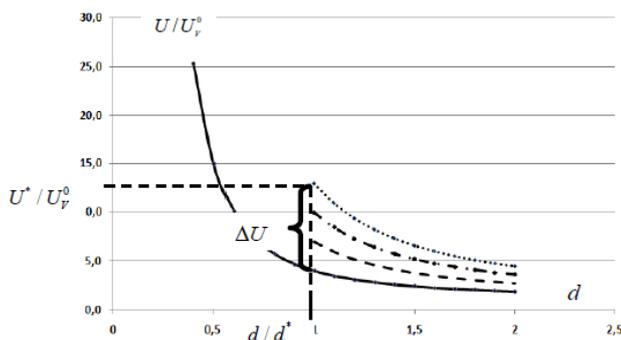


Fig. 5. Dependence of the potential energy from the parameter of mechanical activation process.

Dotted line show process with parameters $U_v^{(0)}, a_1^{(1)}, a_2^{(1)}, a_3^{(1)}$. Solid line corresponds to process with parameters $U_v^{(0)}, a_1^{(2)}, a_2^{(2)}, a_3^{(2)}$. The energy jump ΔU describes the energy releasing due to diffusion process. Dashed-dotted line and dashed line demonstrate increasing of the degree of saturation of nickel in the solid solutions which lead to decreasing of emissions of heat in the acts of diffusion.

The energy releases in the form of heat in the result of the diffusion process. $\Delta U = Q^*$ (see the gap in the Fig. 5.). The magnitude of the potential energy jump ΔU depends on the volume fraction of nickel nanoinclusions passed into solid solution and the degree of saturation of the aluminum with nickel to the initial of the diffusion process. It should be noted that after each annealing step original nanocomposite structure in the vicinity of the phase boundary Ni - Al changes its properties, as on the one hand changes the properties of the matrix, which plays the role of a solid solution, and the other hand the concentration of nickel nanoparticles is varied.

The dotted curve in the Fig. 5 shows the first act of annealing, when aluminum is still under a pure state, and there is no dissolved nickel. In this case the greatest value of heat

energy is released as the largest volume of nickel nanoparticles can participate in the formation of the solid solution, and consequently, a large part of the surface energy of deformation can be released.

Thus, with every act the degree of saturation of nickel in the solid solution in the border area increases. The Fig. 5 shows also two more cases (dashed-dotted line and dashed line), which associated with the re-annealing. Here less amount of heat is released due to the fact that the unsaturated solid solution Ni - Al is the primary structure already instead of aluminum. Therefore, in the diffusion process involved a smaller amount of nickel nanoparticles. Lower dashed curve simulates a situation where after several acts of annealing the very low emissions of heat is realized, because on the this stage instead of aluminum the virtually saturated solid solution of Ni - Al is near boundaries in the layered structures of the granules. It is obvious that after realization of some number of mechanical activation processes with intermediate annealing processes (initiation transition to the solid solution) the process of transition to the solid solution will stop since all Al becomes as saturated solution.

Thus, we propose a model that explains the reason for allocating a significant part of the energy due to the transition of the initial nanoinclusions of nickel into solution. In the result the volume fraction of nickel nanoparticles, concentrated near the boundary of Ni - Al is reduced. Obviously, the transition of nickel in solid solution depends on the volume content of nickel in the original system of Ni-Al, degree of saturation of Al solid solution with nickel, a temperature stress. In this scenario, it is obvious that in a finite number of instruments initiating solid solution transition in solid solution stop since all Al becomes saturated solution. It should be noted that for the considered cases the value d_s generally speaking are various. For simplicity in Fig. 5 these points are combined

V. CONCLUSION

1. The internal energy accumulation process during the mechanical activation of nickel and aluminum powder mixture was considered. The mechanism of the primary energy storage accumulation was associated with the nickel nanoparticles formation in aluminum by the diffusion-free mass transfer process. Low level of activation energy of the diffusion process of nanoparticles dissolution with formation of unsaturated solid solution provides the energy release in the form of composite granules spontaneous self warming during the annealing at the temperature of 150 °C.
2. The mechanism of sequential activation composite granules of a series of successive processes during the mechanical activation, which leads to the initiation of chemical interaction with the nickel monoaluminide (NiAl) formation directly in the working area of mechanical activation unit was based.
3. Changes in mechanical activation technological process were suggested, in order to increase the overall processing time with more thin layers of reactive components of composite granules formation of and a large amount of

internal energy accumulation for the successive of-furnace processes of solid materials formation.

4. The model of low-temperature activation processes is proposed. A qualitative explanation is given for the initiation process of intermetallic formation chemical reaction at low temperature activation. A physical explanation is given to the phenomenon of allocation of thermal energy during the intermediate annealing and its role in the management of low-temperature synthesis. The connection between the level of released heat and the system strain energy state and structural characteristics was established.

ACKNOWLEDGMENT

This work was carried out with support in part from the Ministry of Education and Science of the Russian Federation in the framework of Increase Competitiveness Program of NUST "MISiS" No/ K3-2014-068 and with the support from the Russian Foundation for Basic Research Grant 12-01-00273-a.

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Nb with Al_2O_3 reinforcing, obtained by the mechanochemical synthesis.



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