

New results on structural macrokinetics obtained on multilayer nanofoils

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Abstract. SHS process in the multilayer nanofoils possesses many unique properties, such as unexpectedly high propagating rate (up to 10^2 m/s) and extremely short time of reaction and product phase formation (10^{-7} – 10^{-6} s). Understanding of the mechanism of this process has critical significance not only for the theory of SHS, but also for various applications of the reactive nanofilms, e.g., joining of dissimilar materials and items. An overview of new experimental results shows existence of complex structure of the reaction waves in the multilayer foils and reveals some new “solid-flame” combustion phenomena at macroscopic level. Comparison of these data with the study of the micro- and nano-scale processes by different experimental methods sheds new light on the intrinsic mechanism of the process. The roles of the melts, reactive exothermic dissolution and metastable phases are discussed.

Introduction

Reactive multilayer nanofoils (RMNF), most commonly produced by means of altering layer-by-layer magnetron deposition on the cold substrate, stand out against other multilayer thin films due to their ability of self-sustained exothermic reaction between the layers [1,2]. Thickness of the layer may vary from several nanometers to microns, while number of the layers “stacked” in one foil may achieve several thousands. The overall thickness of the foil is limited by mechanical stresses accumulating in the heterogeneous lamellar structure composed of dissimilar materials, and usually do not exceed 200 micron. Although the Ni/Al MRNF’s are the most studied system of numerous experimental and theoretical works, many other Me/Al bimetal nano-foils, where Me = Ti, Co, Pd, Pt, Cu, Nb, Ta, were obtained using this method [2]. Besides, some metal/nonmetal MRNF’s have been produced recently by magnetron sputtering, including Nb/Si [3] and Ti/2B [4].

General limitations of the magnetron sputtering are the high cost and relatively low yield. The quest for alternative, efficient production methods led to development of the multiple cold rolling technique [5-9], also named as accumulative roll bonding process [10,11]. Bimetal stack of alternate foils undergoes cold rolling several times, which results in reduction of the initial thickness by the order of value or more. For example, using stack of original Ni (18 μ m thick) and pure Al (25 μ m) foils, the multilayer foil with bilayer thickness of 5 μ m, total thickness of 0.85 – 0.9 mm was obtained [6]. Modification of this method involves hot rolling of the bundle of metal sheets (foils) in vacuum at 750 – 1000 °C as a first technological stage, which provides formation of multilayer bulk material [12]. Instead of hot rolling, hot vacuum pressing can be applied at the first stage for the same purpose of producing bulk multilayer rolling billet [13]. The second stage is multiple cold rolling of this material in air. It was revealed that the “hot + cold” rolling technique

facilitates the formation of nanolaminate bimetal composites with layer thickness down to a few nanometers, and number of layers up to tens of thousands. However, the real nano-laminates were obtained in the systems of nonreactive, immiscible metals. Thus, multilayer foil with the flat continuous nano-layers and advanced mechanical properties was obtained in the Cu-Nb system: bilayer thickness was about 10 -20 nm [14]. Nanolaminate composites with tortuous, nonuniform layers were reported for the Cu-Ag system with limited solubility [15]. For the reactive systems, with ability of the intermetallic phases formation, the thickness of individual metal layers in the laminate shoots up to several microns: the microstructures presented in the literature possess bilayer period over 50 μm for the Cu-Ti [13], about 20 – 30 μm for the Ti-Ni [16], and 140 μm for the Ni-Al [17] composites. Therefore, mechanical methods allow production of nanometer-scale laminates in the nonreactive immiscible systems, and micrometer-scale laminate composites of the reactive metals.

Non-uniformity, intermittence and tortuosity of the layers are the most common microstructure defects of the reactive multilayer foils produced by mechanical methods. Commonly, the thickness of individual layers varies in the range 0,1 -10 μm . These effects are probably caused by difference in mechanical properties of Ni and Al (yield strength, hardness, etc.) and sliding of the layers relative to each other. In order to improve contacts between layers, careful cleaning of the surfaces of initial foils is used shortly before the cold or hot rolling, e.g., wire-brushing and cleaning with acetone for the cold rolling [8], grinding with abrasive paper for the hot+cold rolling [13], etc. The pre-treatment of the initial metal foils or sheets removes surface oxides and other impurities, and creates proper surface geometry for better adhesion of the layers.

The third branch of production methods, which should be noted here, is a combination of high-energy ball mill and cold rolling. The idea of this combination, suggested in [2], has been accomplished as a technological method in [18]. The ball milled and cold rolled foils possess relatively high non-uniformity, intermittence and tortuosity, as compared to the magnetron deposited films. However, reactive properties of the foil depend mostly on the thickness of the reactants layers, while their shape has negligible effect on the reactivity.

Experimental results and discussion

Kinetics of the heterogeneous reactions strongly depends on the temperature – time regime of the process. Most of the systems under consideration react at growing temperature conditions, when the temperature rises due to external heating or internal reaction self-heating. All possible reaction stages and intermediate products can be revealed at slow heating, e.g., using Differential Scanning Calorimetry (DSC) method, where the testing samples are heated at the rate of 0.1 – 1 K/s. The reaction route changes dramatically, when the heating rates increases up to 10^5 – 10^7 K/s, which are typical parameters of the combustion wave front. When the heating rate is very high, most of the reaction stages and products do not occur; the reaction mechanism is simplified and often reduced to one high-temperature stage. For instance, in the Ni/Al nanolaminates, slow DSC heating revealed the formation of several intermediate phases, including primary phase NiAl_3 , metastable Ni_2Al_9 , and dominant growing Ni_2Al_3 , preceding appearance of the final B2 NiAl phase [19,20]. Direct measurement of the phase transformations during combustion of the Ni/Al multilayer nanofoils, made by time-resolved X-ray micro diffraction using synchrotron radiation, has shown that the reaction occurred in one stage, and NiAl was the only product [21]. Probably, this rule is applicable also for other compositions of the MRNF. A nanocalorimetry method, specially developed for investigation of heterogeneous reaction mechanisms in the nano-layer systems at high heating rates, was applied, in conjunction with modeling, to the Ni/Al foils with 50 nm and 60 nm bilayers [22]. The results have shown one exothermic reaction stage in the temperature range 600 – 1000 K, with the maximum chemical self-heating rate about $4 \cdot 10^5$ K/s.

Two intrigue features of the MRNFs are very low temperature of the reaction initiation and high velocity of the reaction wave propagation (gasless combustion). Compilations of the experimental data were presented in review [2]. Self-sustained reaction can be initiated at temperature of 150 - 200°C, much below melting point of any metal component, and 300 – 500

degree lower than the ignition temperature of micro-heterogeneous powder mixtures with similar compositions. Commonly, solid state diffusivity is negligible at that temperature, however, nano-heterogeneous materials may possess different transport properties. Thus, nanocalorimetry measurements [22] gave the following expression for calculating of inter diffusivity in the Ni/Al nanolayers at temperature $T = 720 - 860$ K:

$$D = (2.79 \times 10^{-10}) \exp\left(-\frac{6900}{T}\right) \text{m}^2/\text{s} \quad (1)$$

Calculation by this formula gives $D = (2,0 \div 9,7) \times 10^{-14}$ m²/s, which is orders of value higher than expected from referenced data on bulk materials for the corresponding temperature range. Molecular dynamics simulation of the process of reaction initiation (self-ignition) in the Ni/Al nanolaminates demonstrated significant role of interphase boundary misfits in the reaction initiation [23]. Increasing of the misfit stress up to 6,87% results in decrease of the calculated reaction ignition temperature from 800 K to 450 K. Another hypothesis on the causes of hyper-reactivity is that thin intermixed layers on the interphase boundary, which form during deposition, serve as precursors of the reaction, lowering activation barrier for the reaction start. Thus, initiation of the self-sustained reaction arises from solid state processes, which occur with unusually high rate at relatively low temperature due to enhanced diffusivity in the nanostructure heterogeneous materials and, possibly, due to nanoscale solid precursors. The mechanisms of these solid state processes are the subjects for the nearest future investigation.

Once initiated, the exothermic reaction releases heat sufficient for sharp increase of the temperature above melting point of at least one reactant. Hence, the main stage of reaction includes interaction between solid refractory metal layers and molten compounds. The solid-liquid reaction mechanism must explain the above mentioned high velocities of the reaction wave propagation, which can reach 80 -90 m/s [2]. One of the existing models presumes that the continuous layer of solid product forms at the interphase boundary, and the reaction proceeds due to diffusion of the reactants through this layer, as shown schematically in Fig. 1a. According to this model, in the Ni/Al reactive multilayer foils, Ni and Al atoms have to diffuse through the NiAl solid layer. Evaluation of the diffusivity obtained as best fits of the experimentally measured reaction wave propagation velocity [24] gave:

$$D = D_0 \exp\left(-\frac{E}{RT}\right) = (2,18 \times 10^{-6}) \exp\left(-\frac{16478}{T}\right) \text{m}^2/\text{s}, \quad (2)$$

where $R=8,314$ J/mol·K – is the universal gas constant, $E=137$ kJ/mol is the activation energy. Despite of the strong differences in the pre-exponent factors and activation energies, calculations by formulas (1) and (2) provide close values of the diffusivity coefficient in the low-temperature region. At $T=860$ K equation (2) gives $D = 1,2 \cdot 10^{-14}$ m²/s, which is about 8 times lower than the result of calculation by equation (1), and still higher than expected from data on bulk materials.

Calculations by using (2) for the high-temperature range led the following values: for the adiabatic combustion temperature (1900 K) $D = 4,0 \cdot 10^{-10}$ m²/s; for the melting point of Ni (1728 K) $D = 1,7 \cdot 10^{-10}$ m²/s; for the eutectic point (1658 K) $D = 1,1 \cdot 10^{-10}$ m²/s. These values are much higher than the diffusivities of the Ni and Al in the B-2 NiAl determined from the independent measurements [25], which can be described for the stoichiometric (50/50 at%) nickel aluminide as

$$D = (3,45 \times 10^{-5}) \exp\left(-\frac{34881}{T}\right) \text{m}^2/\text{s}. \quad (3)$$

The equation (3) was derived from the experimental data in the temperature range 1050 – 1630 K. Extrapolating this equation for higher temperatures, one can estimate the diffusivity of Ni in NiAl at combustion temperature as $4,1 \cdot 10^{-13}$ m²/s, or at Ni melting point as $6,8 \cdot 10^{-14}$ m²/s. According to molecular dynamic simulation, diffusivity of Al in solid NiAl, is about 2.5 times slower than that of Ni [26]. The results show that solid state diffusivity, determined from independent experiments and simulations, is $10^3 - 10^4$ times lower than that is required for the explanation of high gasless combustion velocity within the frames of the model shown in Fig. 1a. One can argue, of course, that diffusivity in nano – materials is higher due to large concentration of crystal lattice defects, grain boundaries and other specific features of the *nano* state of the matter. However, experimental proofs and measurement of the diffusivities in the highly-defective solids are not available yet for the

considered here systems. On the other hand, self-diffusion of Ni in the Al-Ni melt, experimentally measured by means of quasielastic neutron scattering at 1928 K, was found equal to $4,9 \cdot 10^{-9} \text{ m}^2/\text{s}$ [22]. Therefore, if we assume that the reaction is controlled by diffusion in the melt, extremely high rates of the reaction wave propagation find their obvious explanation.

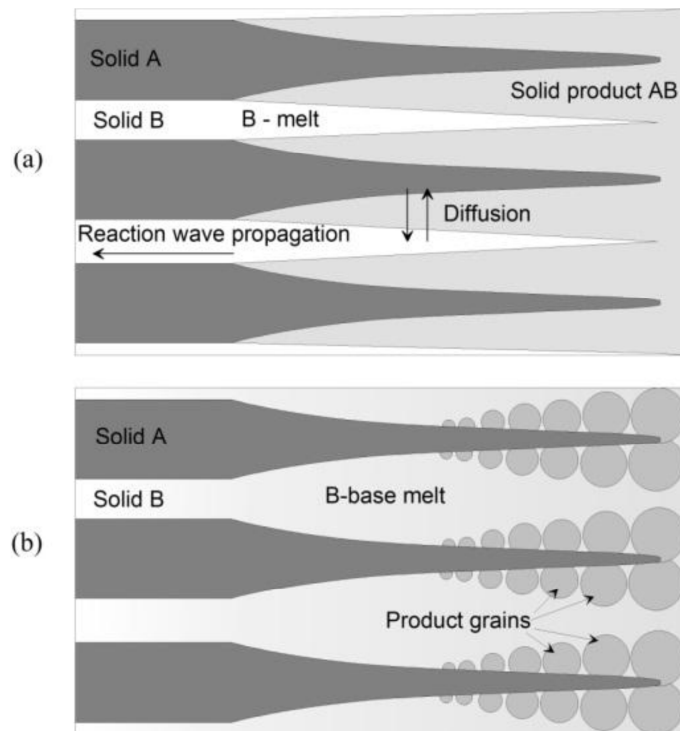


Fig. 1: Microstructure models of the heterogeneous self-propagating reactions in the multilayer nanofoils (a) – solid state reactive diffusion through the continuous product layer; (b) – dissolution into the melt followed by precipitation of separate solid grains.

The reaction model based on direct dissolution of the refractory compound into the melt and liquid-state diffusion, is presented schematically in Fig. 1b. This model assumed that the grains of solid products does not form permanent layer, therefore, liquid state diffusion takes place in the gaps between the grains. This mechanism was experimentally found in the quenched Ni/Al MRNF reaction waves [28]. Microstructure of the reaction zone of the gasless Ni/Al combustion wave, obtained by quenching of the reaction process, is shown in Fig. 2a. It is evident that the solid product consists of separate spherical nanosized grains, which doesn't form continuous solid layer. Similar conclusion had been made from the molecular dynamics simulation of the reaction[29]. Thus, we can make a conclusion that the model of direct dissolution into the melt and precipitation of separate grains adequately describes experimentally observed features of the heterogeneous high-temperature reactions in the nano-layer gasless energetic materials.

Reaction mechanisms in the multilayer foils produced by mechanical methods (cold rolling) are less puzzling. Since the thickness of the layers realized by this method so far lies in the micrometer-scale range, the combustion front propagation velocity does not exceed several centimeters per second [30]. Such values can be explained by solid or liquid state diffusion mechanisms and, probably both of these processes occur in the mechanically structured multilayers.

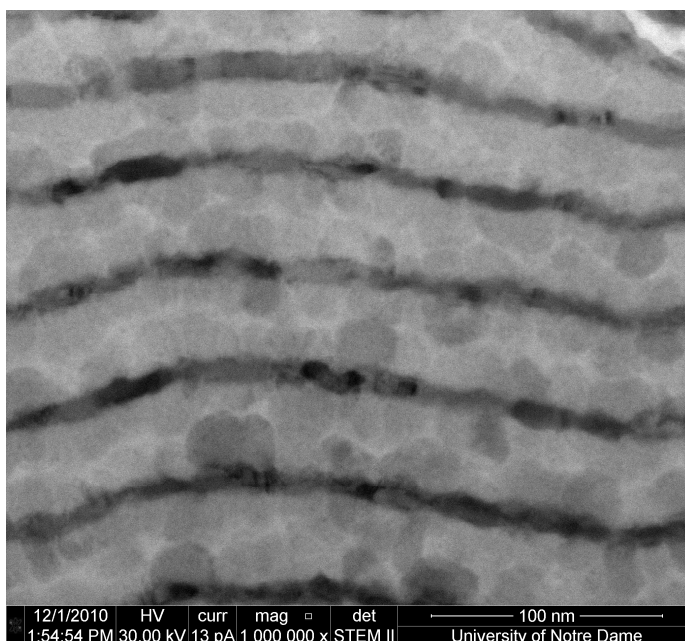


Fig. 2: Microstructure of the reaction zone: quenched Ni/Al reaction wave (TEM; dark phase is Ni, white – Al, grey - NiAl).

Applications of the MRNFs follow from their thermodynamic properties and reaction mechanisms. Due to the high reaction rate and fast heat release, these functional materials find its applications in joining (bonding) materials, in the reaction initiating systems, for enhancing of the heat and blast effects of energetic materials, etc. For the joining purposes, the reaction foil is placed, together with the solder layers, between two materials (items), similar or dissimilar, which are to be joined. The precision amount of heat, instantly released by the foil after reaction initiation, melts the solder layers and joins the materials, without damaging effect on their structure and properties. A wide spectrum of different materials and items, from refractory metals and ceramics to sensitive microelectronic components (e.g., LED crystals) and amorphous metal glasses can be soldered. More detail reviews of this class of applications can be found in [1,2]. Magnetron - deposited Ni/Al multilayer nanofoils are the most used, commercially available soldering composite, characterized by a heat release $\Delta H = 118$ kJ/mol and self-heating adiabatic temperature $T_{ad} = 1911$ K. Some recent examples of the low-temperature bonding in microsystem technology are: IR-emitter was bonded on a covar socket; quartz strain gauge was bonded on a steel membrane [31, 32]. Higher (as compared to Ni/Al) heat release and temperature can be obtained in the Pd/Al ($\Delta H = 163$ kJ/mol; $T_{ad} = 2270$ K) and Pt/Al ($\Delta H = 201$ kJ/mol; $T_{ad} = 2580$ K) systems. Formation of the non-desirable intermetallic phases [33] and relatively high cost of the Pt and Pd hinder R&D of these compositions, however, successful applications have already been reported [34]. Specific feature of the work [34] is that very thin (0.9-4.5 μm) multilayer nanofilms (bilayer thickness 75 – 400 nm) were deposited directly on the components of the microelectronic devices. After deposition, the proper pattern of the reactive film on the substrate was created by wet etching. Successful bonding was obtained using Pd/Al and Ti/ α -Si multilayers.

The second branch of the practical problems, solved by utilization of the MRNFs, relates to the pyrotechnic and military applications, such as enhancing blast effect of energetic materials [10], or as elements of various initiators [35, 36]. Initiation and reaction of the multilayer nanofoils under fast dynamic compression have been studied in different experimental works using several methods of the dynamic loading. Shockless laser-driven compression of the Ni/Al MRNFs was obtained by means of the laser beam pulse with wavelength 351 nm, 3.7 ns duration and intensity $(3,8 \div 7,6) \times 10^{12}$ W/cm² [9]. The laser beam was not focused at the foil surface, but at the intermediate ablator reservoir, located at short distance before the foil. Expanding dense plasma, generated due to laser exposing of the ablator-reservoir, compress the foil without formation of the

shock wave. Fully reacted NiAl with grain size $\sim 500\text{nm}$ was found in the compressed region, however, the exothermic reaction did not become self-sustaining and did not propagate throughout the foil. Similar results were obtained for the direct laser shock-wave loading, when the foil surface was irradiated by the light beam with intensity $2,68 \times 10^{11} \div 1,28 \times 10^{13} \text{ W/cm}^2$, and pulse duration 3 and 8 ns: the laser shock created craters, spall, and extreme damage occurs in the laminates, but did not generate self-propagating reaction through the sample [6]. These results are puzzling, because the self-sustaining reaction in the Ni/Al nanofolios can be easily initiated by weak electric spark, or by local heating up to 200 – 300 °C. Probably, the reason is that the duration of the laser pulse is not enough to form the preheated region comparable with the thickness of preheating zone of the steady reaction wave, which is required for ignition. It is also possible that destruction and fragmentation of the exposed region prevents formation of the preheated zone, because the hot pieces of the foil fly out of this zone.

Different result was obtained using mechanical impact [37]. The flyers made of different materials (e.g., Cu or Al) were accelerated by laser beam, up to velocity 1,4 km/s, and strike the Ni/Al vapor-deposited MRNF. It was found that the reaction initiation threshold impact velocity was 270 m/s for foils with 30-nm bilayer spacing, and that the reaction to the NiAl phase was self-propagating once initiated. Commonly, the shock loading or impact is applied along the normal to the foil surface, and, therefore, normally to the layers. Computer simulation of the shock wave propagation inside the laminates, made using real microstructures of the cold rolled Ni/Al materials, led to conclusion that orientation of the layers can greatly affect the dissipation and dispersion of the shock waves [7].

The electrically exploding foil initiators, due to their safety and reliability, are widely used in military and civilian applications. Additives of the Al/CuO magnetron sputtered MRNF to the exploding foil initiator was studied in the work [35]. It was concluded that the reactive multilayer foil has the potential to decrease the initiation threshold, which will lead to a smaller volume of initiator, further increased safety and reliability. Very simple micro-initiator device was suggested using Ni/Al nanolaminates [36]. The ignition power threshold was found as small as 3 mW with the ignition delay about 0.63 s. Unlike the exploding initiator, the latter device produce only heat, without any mechanical impact, and, therefore, cannot be used for initiation of the explosives.

The gasless exothermic reaction in the MRNF, at some conditions, can initiate solid – gas reaction of oxidation between unreacted components of the same film and gas environment (oxygen, etc.). This solid-gas reaction increases energy output of the reactive material (foil) and duration of the heat release. The potential application of the reaction foil as “self-initiator” in oxidizing environments was demonstrated for the Ti/2B [4] as well as for the 3Al/2Ni, 3Al/Zr, and Al/Zr [38] compositions.

Summary

Finally, it should be noted that most of the considered multilayer reactive nano – foils can be used for the combustion synthesis of fine-grained (sometimes, nanocrystalline) intermetallics. However, taking into account relatively high cost of the MRNF production, this method does not look like economically effective production route. Some of the multilayer foils possess interesting mechanical or physical properties, e.g., high tensile strength or superconductivity. The considered results also show that the reactive multilayer nanofolios represent very promising objects to study macrokinetic mechanisms of the heterogeneous processes under extreme temperature conditions.

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