Features of Electrophoretic Formation of Local Heat Sources Based on Nanosized Powder Al

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Abstract. In this study, the features of electrophoretic deposition (EPD) method to form nano-Al based energetic layers were investigated. The influence of solvents and additive components on the EPD process was analyzed, and the optimal composition of the suspension for the best deposition of layers based on nanoscale Al particles was acquired. The obtained layers can be used as an initiator of secondary reactions for on-chip energetic systems and a local heat source for joining surfaces by reactive bonding.

1. Introduction
Nowadays nanoscale energetic materials attract considerable research interest as a local heat source for low-temperature joining of thermosensitive elements [1]. Another one of the fields where these materials can be used is as an initiator of secondary reactions for on-chip energetic systems. Usually thermit composite materials consist of fuel particles and oxidizer particles [2, 3]. The most represented fuel material is aluminum. Among oxidizers, metal oxides such as CuO, Fe₂O₃ and WO₃ are popular. If using more active nanosized aluminum and initiate combustion in air, then an oxidizer is not needed - oxygen from the atmosphere will be used for the reaction.

In order to obtain such coats electrophoretic deposition (EPD) is considered to be the most affordable method that has the ability to deposit thin layers locally [4]. Thus, goal of this study was to investigate how suspension composition affects its stability and how process modes affects the morphology of the layer surface.

2. Methodical
To carry out the experiments it is necessary to prepare suspensions. They consisted of solvent, main and additive components [5]. For solvents, isopropyl alcohol (IPA) and acetone were used solely and in different ratios together. The main component of the suspension was Al nanopowder synthesized by the method of wire electric explosion in the argon atmosphere at Advanced Powder Technologies LLC. The aluminum powder has the particles with the mean size of 90–110 nm and contains at least 85–87 at.% of aluminum and 13–15 at.% of oxygen, the presence of which is associated with the thin layer of native oxide on the surface of the particles. Additive components as nickel (II) nitrate hexahydrate Ni(NO₃)₂·6H₂O was used for suspension stabilization. The suspensions were dispersed in the ultrasonic bath. All suspension compositions are presented in the Table 1.
Table 1. Suspension composition.

| № | Suspension composition | Al mass, mg | N₂NiO₆·6H₂O mass, mg | Solvent ratio, ml |
|---|------------------------|------------|-----------------------|------------------|
| 1 | IPA/Al                 | -          | 50                    |                  |
| 2 | IPA/Acetone/Al         | -          | 25/25                 |                  |
| 3 | IPA/Acetone/Al/N₂NiO₆·6H₂O | 100       | 10                    | 25/25            |
| 4 | IPA/Al/N₂NiO₆·6H₂O     | 10         | 50                    |                  |

The EPD process requires an electrophoretic cell. It includes two electrodes connected to a power source and a suspension immersed into a beaker. Titanium foils were used as substrates on which the material was deposited, and stainless steel plates were used as a counter electrode. The area was masked with round template, deposited surface area was 1.5 cm². Layers were deposited at potentiostatic mode at an electric field strength from 30 to 150 V/cm at different time limits.

Surface morphology, thickness and stoichiometry of the obtained energetic materials were investigated using scanning electron microscopy (SEM) with an attachment for analysis of energy dispersive X-ray spectroscopy (EDS).

3. Results and discussion

First, the stability of suspension has been investigated. To stabilize it different solvents and additives were used. The change in transparency and the formation of sediment at the bottom of the tube as well as uniformity of deposited layer were visually assessed. Within the framework of this work, a suspension was considered stable if the time of its visual stability was many times greater than the duration of the electrophoretic deposition process.

First, layers obtained from the suspension composition № 1 have uneven surface, because stability of suspension was low (Figure 1, a). Visually suspension sedimented on the bottom of the beaker in approximately 10 minutes. Then it was decided to add acetone to the suspension (composition № 2, Table 1). Visually layers were uniform, which tells about higher stability of the suspension in general (Figure 1, b). Afterwards, composition № 3 was tested. Layers obtained from the suspension have visually bigger deposited surface area although it was masked (Figure 1, c). Therefore, after removing acetone from composition № 3 layers were more even (Figure 1, d).

Figure 1. Photos of specimens obtained from suspension composition presented in table 1 respectively: (a) – composition № 1, b – composition № 2, c – composition № 3, d – composition № 4.

According to the experimental results, two compositions of suspensions showed better stability – suspension composition № 2 and № 4 (Table 1). The addition of acetone into the suspension made of Al and isopropyl alcohol allows to increase the stability of the suspension. In another case, the addition of nickel (II) nitrate hexahydrate to the same suspension composition makes it possible to stabilize it. In both cases layers were visually uniform.

Afterwards, the influence of different concentrations of N₂NiO₆·6H₂O to the velocity of the EPD process was studied (Figure 2). There were 4 suspension compositions with different concentration of N₂NiO₆·6H₂O (Table 2).
Table 2. $\text{Ni}_2\text{NiO}_6\cdot6\text{H}_2\text{O}$ concentration in the suspension.

| Suspension composition | Al mass, mg | $\text{Ni}_2\text{NiO}_6\cdot6\text{H}_2\text{O}$ concentration, mg/ml | Solvent volume, ml |
|------------------------|-----------|-------------------------------------------------|------------------|
| IPA/Al/$\text{Ni}_2\text{NiO}_6\cdot6\text{H}_2\text{O}$ | 100       | 0,1                                             | 50               |
|                        |           | 0,2                                             |                  |
|                        |           | 0,3                                             |                  |

Layers were deposited at potentiostatic mode at an electric field strength from 30 to 150 V/cm with 20 V/cm increments. Consequently, seven specimens from each suspension were obtained. Analyzing the dependence of the deposition velocity on $\text{Ni}_2\text{NiO}_6\cdot6\text{H}_2\text{O}$ concentration suspension composition with 0,1 mg/ml concentration of the charger has higher velocity. Though it was decided to use the composition of the suspension with 0,2 mg/ml concentration of $\text{Ni}_2\text{NiO}_6\cdot6\text{H}_2\text{O}$, because the stability of the suspension was higher.

![Figure 2. EPD velocity dependence on $\text{Ni}_2\text{NiO}_6\cdot6\text{H}_2\text{O}$ mass.](image)

Though stability of the suspension composition № 4 from Table 1 was good, the velocity of the deposition was lower than in the composition № 2 from the same table. Thus it was decided to use isopropyl alcohol/acetone/Al composition. Graph of the deposited weight dependency on electric field strength has linear tendency (Figure 3). Varying such characteristics as electric field strength and time limits it is possible to control deposition velocity.

![Figure 3. Deposited weight of the specimen dependence on electric field strength, deposited from IPA/Acetone/Al suspension.](image)
Then the morphology of the obtained coats was examined with the scanning electron microscopy. Layers deposited in the electric field of 10 V/cm have big agglomerates and was uneven. The deposition velocity in the electric field of 110 V/cm was higher, thus deposited layers turned out to be more porous, which negatively affected the adhesion of the formed layers, and some areas of the layer desorbed from the substrate during the process. In the course of the research it was revealed that coat deposited at the electric field strength of 70 V/cm is more even than coat deposited at 10 V/cm and less porous than coat deposited at 110 V/cm (Figure 4). In this regard, the electric field strength of 70 V/cm was chosen.

![Figure 4. SEM photos of specimens obtained from suspension composition IPA/Acetone/Al.](image)

Therefore, suspension composition of choice is isopropyl alcohol and acetone 1:1 ratio (50 ml), Al (100 mg); EPD mode – 70 V/cm, 5 minutes.

After finding out the electrophoretic deposition mode, examination of self-propagating high-temperature synthesis. The self-propagating reaction was initiated by a lighter. Combustion was propagating evenly through the entire surface area. Complete combustion of a double-sided specimen with the length of 3 cm continued for 4.3 seconds. Combustion rate frames were acquired using VirtualDub software (Figure 5).

![Figure 5. Combustion rate frames.](image)

Knowing length of the specimen and time of self-propagating high-temperature reaction the combustion rate was calculated. It is appeared to be 0.007 m/s.

4. Conclusions
Based on the research results, the influence of solvents and additive components of suspension on the EPD process was analyzed, and the optimal composition of the suspension for deposition of layers
based on nanosized Al particles was acquired. The obtained coats can be used as an initiator of secondary reactions for microelectromechanical systems (MEMS), and as a local heat source for reactive bonding of the surfaces.

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