NANOFOLIJE ZA LOTANJE PRI ZOBOZDRAVNIŠKEM DELU IN IZDELAVI NAKITA

Karlo T. Raič1, Rebeka Rudolf2, Borut Kosec3, Ivan Anžel2, Vojkan Lazić4, Aleksandar Todorić4

1University of Belgrade, Faculty of Technology and Metallurgy, POB 3503, 11120 Belgrade, Serbia
2University of Maribor, Faculty for Mechanical Engineering, Smernetova 17, 2000 Maribor, Slovenia
3University of Ljubljana, Faculty of Natural Sciences and Engineering, Aškerčeva 12, 1000 Ljubljana, Slovenia
4University of Belgrade, School of Dentistry, Clinic for Prosthodontics, 11120 Belgrade, Serbia
karlo@tfm.bg.ac.rs

This paper describes the development of novel, reactive Al-Au nano-multilayered foils, their possible application in different fields and a discussion of the nano-folios’ suitability for dental and jewellery applications. Moreover, this study includes the rapid joining of similar and dissimilar materials, by placing multilayer nano-folios and two layers of solder or braze. The foils precisely control the instantaneous release of heat energy for the joining and act as a controllable local heat source. The reactive foils’ thickness is in the range 10 nm to less than 100 nm and they contain many nanoscale layers that alternate between materials with high mixing heats, such as Al and Au. The foil between the two solder/braze layers melts the solder/braze with the heat generated by the reaction and bonds the components. The use of reactive foils eliminates the need for a furnace and dramatically increases the soldering/brazing heating rate of the components being bonded. Thus, ceramics and metals can be fused over required areas without the thermal stresses that are encountered in furnace soldering or brazing. In addition, a completely new plasma technology is proposed for the manufacturing of nano-folios and the first results of the preliminary experimental testing are presented.

Key words: ceramic-metal bonding, low-temperature, nano-folios, plasma technology, dentistry, jewellery

Članek opisuje razvoj nanoplastnih reaktivnih Al-Au-folij, njihovo uporabo na različnih področjih in razprava o uporabi v zobni tehniki in pri izdelavi nakita. Studija vključuje hitro spajanje podobnih in različnih materialov s postavitvijo večplastnih nanofolij ter plasti lota med oba dela za spajanje. Folije kontrolirajo hipno sprostitev toplotne energije za spajanje in delujejo kot lokalni vir toplote. Reaktivne folije z debelino od 10 nm do 100 nm so izmenoma iz materialov, ki imajo veliko toplotno mešanja, npr. Al in Au. Folija z reakcijsko toploto raztala obe plasti lota in spoji obe lotani komponenti. Pri uporabi reaktivnih folij je peč nepotrebna, zato se zelo poveča hitrost spajanja lotanih komponent. Keramiko in kovino je mogoče spajati na določenih površinah brez toplotnih napetosti, ki nastanejo pri lotanju s segrevanjem v peči. Sugirana je tudi nova plazemska tehnologija za izdelavo nanofolij in predstavlja so prvi rezultati praktičnih preizkusov.

Ključne besede: spajanje keramika kovina, nizka temperatura, nanofolije, plazemska tehnologija, zobotehnik, nakit

1 INTRODUCTION

Multilayer structures are thin-film materials that are periodic in one dimension with regard to composition or structure. Composition/structure variations are generated during the structure’s synthesis, which is typically accomplished using atom-by-atom, atom-by-molecule, or molecule-by-molecule technologies. Multilayer structures can be synthesized using elemental, alloy, or compound layers to form both microstructures and combinations of elements/materials that cannot be produced using a traditional processing technology. Multilayers are made by the alternate deposition of two or more different materials. After the first few layers, the structure of all the layers of one material is the same. The structure of each material is clearly of importance for the properties of the multilayer, not only in itself, but also for the influence it can have on the structure of the other material. Each material acts as a substrate for the deposition of the following. The simplest multilayer structures are those that consist of a composition modulation imposed on a single structure. In almost all cases of this type, intermixing can lead to a uniform single phase of the starting crystal structure or atomic geometry 1–3.

Nanoscale multilayer foils are usually produced with physical vapour deposition or magnetron sputtering of hundreds to thousands of alternating layers, with layer thicknesses of approximately 1–102 nm. The process of bonding with reactive multilayer foils is very rapid and easily performed in air (there is no need to join the bonds in a clean room or a specially designed furnace). However, it requires a certain preparation of the components. Most typically, layers of solder or braze are pre-applied to the bonding surfaces of the components. It is also possible to use free-standing sheets of solder placed between the reactive foil and the components, provided the surfaces of the components are easily wetted when...
K. T. RAIĆ ET AL.: NANOFOILS FOR SOLDERING AND BRAZING IN DENTAL JOINING PRACTICE ... using the solder or braze. This implies that most ceramics’ surfaces need to be metallised. Following this preparation, the reactive foil is placed between two components that are aligned and a load is applied. In order to ensure good contact between all the surfaces during fusing, the required value will vary depending on the roughness and flatness of the bonding surfaces. The reaction is then initiated by an electrical or optical pulse, heat is released, the solder or braze reflows, and a bond is formed in approximately one second. For large-area bonds it is common to use multiple, simultaneous initiations of the foil to ensure uniform melting of the solder or braze layers across the bonded area. A number of large-area bonds have been created using reactive foils as the local heat source. Localized heating of the reactive foils enables the formation of large-area ceramic-metal bonds by minimizing the residual stresses between those components having appreciable differences in CTE. The transfer of heat from the reactive foil into the adjacent components during a bonding operation has been numerically modelled and the results experimentally validated using infrared measurements. The numerical modelling is performed using a numerical scheme that is based on finite-difference discretization.

The shear strengths of various ceramic-metal configurations joined with reactive multilayer foils and different solders or brazes are available in the literature. It is important to know that the measured strengths depend primarily on the strength of the solder used and not on the combination of materials being bonded. Finally, the significant localized heating that occurs during the bonding process enables these types of bonds.

There are an unlimited number of ways that a nano-foil may be applied. Currently, the biggest markets are energetic (propellant ignition) and large-area joining (armour mounting, structural joining, and the joining of sputtering targets), and small-area joining applications such as sealing and component mounting. A nano-foil could be used to join a broad range of components consisting of metals, ceramics, semiconductors, and polymers. Bonding can be performed in many environments and can be completed in a second or less. Because the heat is localized, components can generally be bonded without thermal damage. Nano-foils can be manufactured by the vapour deposition of hundreds of nanoscale layers that alternate between elements, such as aluminium and titanium. By thermally pulsing one end of the resulting foil with energy, the nanoscale layers begin to mix and release heat to the surrounding foil. This leads to more chemical mixing in the joining areas of the foil and a chemical reaction that self-propagates across the full length of the foil.

The reactive nano-foils can be used for producing new types of protective coatings, for joining materials and for creating free-standing membranes on the base of intermetallic or ceramic compounds. Taking into account that there are at least several hundred systems for which self-propagating reactions have been realized in powder mixtures, the few reactive multilayer systems studied so far represent a small part of a wider variety of reactive multilayer films that might be considered as potential subjects for scientific and technological study.

Several combinations of reactive elements and alloys have been tested up to now, including Al/Monel, Nb/Si, Ti/Al, CuO/Al. They all exhibited high propagation velocities of the exothermic reaction waves, up to 15–20 m/s, which indicated the high chemical reactivity of the nano-foils.

In this paper, a completely new plasma technology studied experimentally for the first time, is suggested for the manufacturing of Al–Au nano-multilayered foils.

2 POSSIBILITIES AND APPLICATIONS OF Al-Au NANO-MULTILAYERED FOILS

Al-Au nano-multilayered foils could find two completely new applications in the areas of dentistry and jewellery.

(1) The first application would be in dentistry, because of the following problems that occur in dental joining practice:

(a) Porcelain Fused to Metal Restorations (PFM) – the bond between an alloy (Co-Cr-Mo, Au-Pt) and porcelain veneer

Although dental alloys have different micro-alloying elements (Ir, In, Rh, Nb, etc.), which are necessary for the ability to bond ceramics to the metal’s surface, there are still problems within metal-ceramics bonding. A potential for failure exists through the thick and brittle oxide layer with many alloy systems, because the extent of the oxide formation cannot be readily controlled. A nano-foil placed between the metal and ceramic layers could strengthen the metal-ceramic bond and enable high quality and longer life for the PFM restorations.

This not only represents an overall benefit to the quality of the dental patients’ health, but also long-term savings in health-care-related costs.

(b) Complex Fixed-Removable Restorations – Joining the abutment (an alloy Co-Cr-Mo, Au-Pt) to the precision attachment (female part) and joining a precision attachment (male part) to the denture base acrylic resin (polymethylmetacrylate)

Retainers (usually precision attachments) for Removable Partial Dentures (RPD) are used for complex fixed-removable restorations. Prefabricated attachments have two components – one incorporated in the abutment and the other part of the RPD. Both extracoronal and intracoronal attachments are available. Resin bonding has been used for the retention of the extracoronal attachments. There is some doubt that the retention obtained is adequate for preventing the dislodgement of the attachments.
Intracoronal attachments typically consist of a precision-milled male-female assembly and they are made of Pt-Pd alloys that can withstand those high temperatures associated with the casting of PFM alloys. The female part of the attachment is incorporated in a wax pattern. After the wax elimination, the restoration is cast directly onto the attachment. The male part of the attachment is either soldered to the frame or attached to the acrylic denture base with auto-polymerizing resin. Most dental technicians prefer to solder the parts of the attachment. However, there are failures of attachment systems, especially when they are used with distal extensions. In order to strengthen the bond between the female attachment and the abutment, and the male attachment and the denture base, a nano-foil could be placed in-between.

(c) Dental Implants – the bond between an alloy (cpTi, Ti-6Al-V, Ni-Ti) and Hydroxyapatite, Zirconium-oxide coatings

Dental implants made of cpTi or different Ti-alloys (Ti-6Al-4V) have been used in dentistry for many years. The clinical use of various coatings (plasma-sprayed hydroxyapatite-HA coatings, diamond coatings, diamond-like-carbon coatings, yttria-stabilised zirconia, ZrO2-3 % Y2O3 coatings) on Ti-implants has been widely adopted because the coatings can achieve firm and direct biological fixation with the surrounding bone tissue. They have great potential for use as biomedical implants because of their very high hardness, excellent wear resistance, low friction and bioocompatibility characteristics. However, the poor bonding strength between the various coatings and the Ti alloy has been of great concern for long-term clinical applications.

HA/yttria-stabilized zirconia (YSZ)/Ti-6Al-4V composite coatings that possess superior mechanical properties to conventional plasma-sprayed HA coatings have been developed. The bonding strength is better than that between a plasma-sprayed HA coating and Ti-6Al-4V, but this bond strength could further be improved by placing a nano-foil between an implant and a coating.

A nano-foil placed between an implant (Ti-6Al-4V) and an HA and/or ZrO2 coating could strengthen the bond between the HA/ZrO2 coating and the Ti alloy, and could enable high quality and a longer life for the dental implant.

The other possibility is to find a suitable nanostructured metallic-ceramic coating that could provide a nanocrystalline metallic bond at the interface to the hard ceramic bond on the surface.

(II) The second application in jewellery:

(a) Metal-Stone Joining. The joining of many different materials is necessary in jewellery manufacture (the metal frames of rings, earrings, necklaces, etc. with different kinds of stone – precious, semi-precious, synthetic stones, coral, and pearls).

One of the phases in jewellery manufacturing is placing the precious stone into the metal frame. The stones are initially cut into the desired sizes and shapes, and then polished. The metal frame cast is ground and polished and the stones are joined to it by adhesives, soldering, or by mechanical clamping. The attachment between the stone and the metal becomes weaker over time, because of elastic deformation. If the stone is soldered to the metal frame, the joint between the stone and the metal is stronger. Different stones have different mechanical and physical properties and it is necessary to find an appropriate combination of solder components for each stone. For example, sapphire is an Al2O3 mineral. Sapphire can be soldered after bonding or brazed without bonding. If the sapphire is brazed to titanium without bonding, an alloy with 72 % Ag could be used. Diamond could be brazed to steel with L-Ag40Cd, L-Ag30Cr and L-Ag20Si.

There are two groups of precious stones according to their price: first-order stones (diamond, ruby, sapphire and emerald) and second-order stones (aquamarine, topaz, zircon, onyx, quartz topaz, etc.). Many of them have similar chemical compositions, but their colours are different. The chemical composition of diamond is 99.95 % carbon. Emerald is a green mineral made of aluminium oxide, and its colour comes from chrome or vanadium. Sapphire and ruby are minerals of Al2O3. Sapphire could have a blue colour from titanium and iron, and violet from vanadium. Ruby could have a red colour from chrome or a chestnut colour from chrome and iron. The chemical compositions of the stones could be very important when joining different kinds of stones. Many stones in the second group are very sensitive to heat. The result of heating the stone above its critical temperature could be a change in the colour. Turquoise has a critical temperature at 250 °C, when its colour then changes from sky blue to green. At the critical temperature interval of tanzanite (from 400 °C to 500 °C), the nuances of yellow gradually disappear and the blue colour is intensified. Other precious stones can also exhibit changes in their colours (quartz, topaz, amethyst, aquamarine, etc.). Therefore, it is very important that during the joining of stones to the metal frame the stones are not heated above their critical temperatures. This can be achieved by local heating of the nano-foil that is placed between the stone and the metal frame. Also, a nano-foil placed in between could strengthen their joint and enable a high-quality jewellery piece.

(b) Metal-Metal Joining. Many different pieces of jewellery have several parts included in their construction. These components are usually joined by gold brazing alloys. The rule, that the brazing alloy must have the same purity (in carats) as the pieces that are joined, has to be respected. The colour of the solder must also be the same. It is necessary for the brazing temperature to be higher than the liquidus temperature of the
brazing alloy, but lower than the solidus temperatures of the materials that have to be joined.

The composition of the brazing alloys used for jewellery applications is Au-Ag-Cu, with the addition of metals such as zinc and cadmium. Cadmium is very toxic, especially its vapour, during the melting process, and must be replaced with non-toxic elements. Cadmium could be substituted by the following metals: tin (Sn), indium (In), gallium (Ga), and zinc (Zn). Also, some solders without cadmium have very high concentrations of Ni (5 to 9 %). According to the EU directive 76/769 EES (January 2000), the concentration of nickel in jewellery cannot exceed a maximum of 0.05 %. This EU directive is a prerequisite for the prevention of allergic reactions caused by nickel. A nano-foil, with properly selected solder or braze components (without Ni) placed between metal parts, could prevent allergic reactions to nickel and could ensure the high quality of metal-metal joins.

3 EXPERIMENTAL PROCEDURE

The Al-Au multilayered foil was obtained by plasma-assisted sputtering in a Plasmait® machine. Atoms of a Au-alloy and Al were sputtered from two separated targets/wires (with diameter \( \phi = 3.5 \text{ mm} \)), pure aluminium \( (\omega = 99.99 \%) \), and 14-carat gold alloy with the chemical composition (mass fractions \( w \)): 58.50 % Au, 28.95 % Ag, 12.45 % Cu), Figure 1.

The Plasmait® machine, with its dynamic sealing system, allows for the continuous processing of materials. The wire or tape guided through the machine is processed with a Special Type Glow discharge working within the pressure range 1–10 mbar. Most applications are processed with \( \text{N}_2 \), but Argon was added to the discharge as was the case with the multilayer deposition of an Au-alloy and Al for a better sputtering effect.

The wire speed through the processing chamber while processing the thin films was 30 m/min, which equals \( 0.5 \text{ m/s} \), and the processing power coupled into the plasma was approximately 250 W for the Au-alloy wire and 800 W for the Al wire. All these parameters (pressure, power in plasma, gas composition, wire speed and wire type) were optimized during the processing.

A substrate of pure copper with dimensions 10 mm × 10mm (thickness about 0.5 mm) was placed on a holder, to be exposed to fluxes of atoms from a Au-alloy or Al atoms, consecutively. The required number of alternating layers was obtained by adjusting the number of turns and thickness for a varying wire speed and magnetron source power.

The Cu/Au-Au-Al-Al/Au/ sample was prepared with Al and Au layers with thicknesses of about 100 nm to 400 nm. The substrate temperature was kept below 323 K in order to prevent any diffusion and reaction during the deposition.

The microstructural characterisation of the produced nano-foils was carried out with scanning electron microscopy (SEM-Sirion 400 NC) and energy-dispersive X-ray (EDX) analyses (Oxford INCA 350). For the electron microscopy the polished specimens were put in the chamber of the SEM microscope, with a vacuum of 10⁻⁶ mbar. The microscopic analysis includes an examination of the polished surface and qualitative and quantitative micro-chemical analyses at characteristic points on the interface between the Au-Al nanofoils and the substrate Cu. We observed surfaces using an electron-beam voltage at 30 kV, and over different working distances (6.5 mm, 7.2 mm, etc.).

Finally, the nano-foils were characterised using a Quanta 200 3D environmental scanning electron microscope equipped with a focussed ion beam (FIB), which enables the cleaning and etching of a sample’s surface, transverse cutting directly from the external sample surface, polishing of the cutting surface, and high-resolution observations of the samples’ microstructures.

4 RESULTS

The microstructure of the Al-Au multilayered foils is shown in Figure 2. From the upper view (Figure 2a), a very porous surface could be seen for the last Au nano-foil. This assumption was additionally confirmed by further investigation, namely on the transverse cutting surface (Figures 2b, 2c), where regions could be found with small and large voids over the cross-sections of all four foils. Voids within the single foils are surrounded with the agglomerate. It seems that the atoms from the Au-alloy accumulated non-homogeneously on the surface of the Cu-substrate, and caused the formation of a very porous microstructure. Detailed microstructure observations demonstrate that the foils mainly possess a columnar microstructure. This kind of microstructure is typical for foils deposited on a cold substrate. Precise measurements of each foil discovered that the Al-foils have an approximate thickness of about 100 nm, while for the Au-foils the thickness was about 400 nm. Consequently, we could conclude that at similar technical
parameters of the plasma process, the thickness of the Au-foil is twice as large as for the Al-foil.

EDX line-analyses (Figure 3) over the cross-sections of Al-Au-Al-Au foils also indicate the presence of Ag and Cu atoms in the Au-foils. These elements rise from the alloy, which was used as a wire-positive electrode in the plasma device. The distribution of the elements in Au-foils followed the chemical composition of the wire, which means the higher mass fraction (%) of the element causes a higher peak in the EDX spectrum. On the other hand, only the presence of Al was established for the Al-nano-foil.

The transformations of the pure metals to Al–Au intermetallics at the Al/Au alloy interfaces were not found and interdiffusion between the Cu substrate and the first Al layer was not detected.

5 DISCUSSION

This paper presents nano-foils that were produced using the plasma process. The plasma is an ionised
In a plasma device, the electric field accelerates the ions towards the wire surface (the positive electrode) and electrons towards the edge of the heating chamber (the negative electrode). On their way to the opposite electrode, the particles collide with other atoms and molecules. The less they collide (or the less interrupted their journey), the faster they accelerate and the larger their impact on the wire surface. The plasma device’s heating chamber is filled with a low-pressure inert gas to prevent a chemical reaction between the gas and the wire. The wire is fed through the sealing system and the heating chamber continuously, thereby exposing the wire surface to ion bombardment. The effect of such bombardment is threefold: (1) efficient heating, (2) surface smoothing and (3) effective surface cleaning.

(1) Colliding ions release their energy on the surface and heat the wire. In the heating chamber the ions are directed to the wire surface, while the electrons head towards the outer wall of the heating chamber. As the ions are approximately 50,000 times heavier than the electrons, they make a considerably higher energy impact on the wire than that caused by the electrons on the chamber wall. In fact, the energy conducted to the chamber is negligible compared to the energy conducted to the wire. Effectively, all of the power conducted into the plasma is transferred to the wire (and not the walls of the heating chamber). The unique benefit of the plasma treatment is the concentrated impact on the wire surface (directional and focused heating) resulting in a very efficient heating process. As with the microwave oven, the heating chamber remains cool during the process. Less heat transmission to the surroundings also means a friendlier working environment. The fact that the heating process concentrates on the wire alone makes a plasma treatment very effective for high-temperature applications, such as heat treatment. Moreover, the plasma-assisted heating process can be easily and instantly regulated through the electric potential in the heating chamber. The intensity of the wire treatment can be adapted to the desired value within milliseconds by adjusting the voltage in the heating chamber. In practice, this leads to another benefit of plasma technology.

(2) Plasma treatment results in a smoothing effect on the wire surface. Like lightning striking a tree in a field, ions are directed to the tips of the irregularities. The density of ion bombardment is highest on the sharp edges and nubs on the wire surface, concentrating on the tips of the irregularities until they melt away. This leads to a smoothing effect, causing a reduction in the macro-roughness and an increased micro-roughness of the wire surface. In some cases a wire with a damaged or rough surface can be improved in quality, and can even reach the standards of a magnetic wire surface.

(3) The plasma treatment is also an effective cleaning process. The ions bombarding the wire break down dirty deposits and oxide layers on the wire surface. One can imagine the cleaning process as sand blasting on an atomic scale. A plasma treatment can, therefore, replace the deoxidising, degreasing or other wire-cleaning processes, whilst eliminating the chemicals from the wire-production process. The dirt blasted away burns into ashes and falls off from the surface. The ashes collect at the bottom of the heating chamber and in the vacuum pump’s filters. The materials where plasma technology is less competitive are those that require long annealing times, in other words those that need to be exposed to high temperatures for a long time. For such an application, high-speed in-line annealing is likely to be unsuitable, due to the required length of an inline cooling system, necessary for such an application.

For the production of nano-foils in the first step we used the cleaning and smoothing process in order to ensure a high-quality surface for the Cu substrate (reduction in macro-roughness). With this step we wanted to obtain a surface with a low degree of surface defects, which consequently could lead to the formation of a very homogeneous formatted nano-foil. Based on microstructural observations, we could conclude that this step was very successful because the Cu-substrate surface in the cross-section is pretty flat (on the micro-level). In the second step we used the plasma-deposition technology for producing nano-foils. On the basis of the obtained nano-foils we could see that the process, in the case of Al-deposition, is completely different to that of Au-deposition. The Al-foils are thin (about 100 nm in thickness), compact and almost without big voids. The Au-foils are thick (about 400 nm), with a quite different non-homogenous microstructure, including many defects (pores and non-uniform grains within the foil). We believe that the reason for the non-homogeneous microstructure is the presence of alloying elements (Ag and Cu), which influence atom segregation during the plasma process.

Al and Au are face-centered cubic (FCC) metals with the highest packing density. In order to understand the change in thickness (volume) during the formation of Al and Au layers, we can compare the density and atomic packing factor (APF) of the pure metals, Table 1. The amounts of volume that the atoms occupy calculated by the APF are equal for Au and Al, but the density of the Au is nine times higher than that of Al. So, for the same processing conditions (wire speed of 0.5 m/s for Al wire and Au-alloy) it is to be expected that the Au-foils will be thicker.

Table 1: The density and APF of Au and Al

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<tr>
<th>Density, $\rho$/g/cm$^3$</th>
<th>A. P. F</th>
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<tr>
<td>Au</td>
<td>19.33</td>
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<td>Al</td>
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Based on the description, the main further research goals would focus on the following areas:
– the determination of the nano-foils’ properties, such as the mechanical properties, hardness, chemical inertness (e.g., oxidation, corrosion), biocompatibility, bioresistance, and the wear resistance of metal-ceramic and related joints;
– the optimization of different chemical compositions for obtaining novel nanostructured reactive foils;
– the determination of optimal heat treatment (to estimate the optimal time-temperature diagram) for nanostructured foils. Different heat treatments, training and testing of the materials’ functional properties are part of this optimisation;
– the development of suitable methods for fusing a metal with ceramics and/or a metal by inserting nanostructured multilayered foils. The basis would be the already-known methods, but they would have to be modified in order to comply with the specifically requested characteristics for the investigated metal-ceramic/metal and related joints;
– to gain new knowledge about the long-term stability of novel nano-foils;
– to gain a deeper insight into the mutual dependence of the chemical composition, the purity of the nanostructured coatings, the production route and parameters, the microstructure, and the mechanical and functional properties;
– to take a first step towards industrial production;
– to discover new, possible biomedical and other applications for nanostructured films, i.e., wear-resistant orthopaedic implants, etc.

6 CONCLUSIONS

Plasma technology represents a novel approach in the production of nano-foils. Preliminary results of the produced nano-foils with plasma technology show that there are still problems with ensuring the single foil’s constant thickness into the sandwich nano-structure. Moreover, the obtained microstructure of the nano-foil depends, mostly, on the initial chemical composition of the surface wire, used as the positive electrode. Also, the other important conclusion is that with nearly the same technological parameters of the plasma technology, the formatted nano-foils of different initial materials are dissimilar. Finally, the shape and the quality of the final nano-foil depends on the preparation of the substrate surface.

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