# TESTS FOR APPLYING COATINGS BASED ON ALUMINUM AND SILVER ON ALNICO ALLOYS

Damian BAŃKOWSKI, Piotr MŁYNARCZYK, Bartłomiej SZWED, Joanna KOWALCZYK

*Kielce University of Technology, Kielce, Poland, EU, dbankowski@tu.kielce.pl*

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**Abstract**

The paper presents preliminary research on the application of coatings by the PVD method. The coatings were deposited on an AlNiCo alloy with a suitably pre-prepared surface. Two different coating materials were used. Coatings based on pure silver and pure aluminum were applied. The tests included the assessment of the influence of the coating deposition time on the final thickness of the coatings. The samples after the deposition process were specially prepared for observation under the microscope. Scanning microscopy examinations made it possible to assess the connections of the obtained coatings with the substrate.

**Keywords:** PVD, coatings, alnico alloys, scanning microscopy, silver, aluminum

## 1. INTRODUCTION

Vapor deposition coating technology is a process that causes the formation of coating materials on the surfaces of solids as a result of specific exploitation of gaseous, vaporous or plasma states [1-6]. One of the unconventional techniques for producing coatings is the method of diffusion bonding of thin films with the base material, thereby producing coatings [7,8]. One of the coating methods is PVD technology - physical vapor deposition, which consists in the deposition and formation of a coating as a result of a reaction in the gas phase (with or without plasma activation), wherein at least one gas phase component is formed by physical vaporization or sputtering [9-12].

Another method is PACVD, or plasma-assisted chemical vapor deposition. PACVD is similar to chemical vapor deposition CVD, but with plasma activation or reaction enhancement and pyrolysis. Although the basic principle of CVD coating was discovered in the 19th century, it was not until the 1940s and 1950s that interest in the technique as a way to produce thin, hard coating materials began [11-12]. CVD can be used to deposit compounds such as metal borides, carbides, nitrides and oxides, etc.

PVD differs from CVD in that one or more reactants undergo a state transition from a solid to a gas phase in a vacuum chamber. PVD processes fall into two general categories: PVD sputtering and PVD ion plating. A glow discharge plasma is formed when a potential difference is applied between the anode and a cathode, placed in a partial vacuum (~0.1-0.4 Pa) of a given gas. Plasma contains ions, electrons (both of which are charge carriers) and neutral molecules [13-17]. In the case of Ar plasma, the ions are positively charged and move towards the cathode, while electrons (emitted by the cathode) move towards the anode. Both charges have a velocity determined by the strength of the electric field (proportional to the applied voltage), which is greatest near the cathode where the greatest potential drop occurs (this is called cathode drop) [18-22]. Most of the ionization is caused by electron-atom collisions, which mainly take place in the cathode fallout zone. Modern plasma power supplies initiate ionization by applying a momentary (millisecond) pulse of very high voltage (>1 kV) to the cathode, which injects high-energy electrons into the process chamber [18-19]. In such conventional glow discharge plasmas, there is a balanced number of electrons and ions that make up only a few percent of the total species present, with the remainder being neutral molecules and atoms [20-21]. Of these, some reach an excited state and emit a visible or negative glow when adjacent to the cathode drop region. The color of the glow depends on the wavelength of the photon emitted during electronic transitions in atoms, e.g. nitrogen plasma is mostly blue while hydrogen plasma is almost pink. For example, aluminum or silver can be applied by physical vapor deposition. Silver has many uses, including conducting electricity. In order to increase the antibacterial properties of the surfaces of biomedical devices, antibacterial coatings such as silver (Ag), copper (Cu) and silicon (Si) etc. are applied, using coating techniques, in addition to obtaining antibacterial properties, also improve their mechanical properties [22-26]. Compared to other heavy metal ions, silver ions are probably the most useful because they combine high antimicrobial activity with extremely low human toxicity. Many laboratory tests have also been carried out on animals and a strong antibacterial effect of silver-coated medical devices has been observed. In addition, silver has been used for centuries to treat burns and chronic wounds. The antimicrobial effect of silver nanoparticles has been extensively described against a broad spectrum of microorganisms along with the absence of negative effects such as taste, odor and color [27-28]. In addition, silver nanoparticles have recently been used to treat drinking water. In addition, aluminum and silver-based coatings are used to make mirror surfaces.

## 2. RESULTS

The aim of the work was to check how the coating application time affects their thickness on AlNiCo alloys -the family of ferrous alloys or sinters, which, apart from iron, mainly contain aluminum (Al), nickel (Ni) and cobalt (Co). Have special mechanical properties: high brittleness and relatively high hardness [29,30]. The aluminum (Al) and silver (Ag) coatings in this study were deposited by PVD physical vapor deposition on the Nanomaster NPE-4000 device (**Figure 1**).

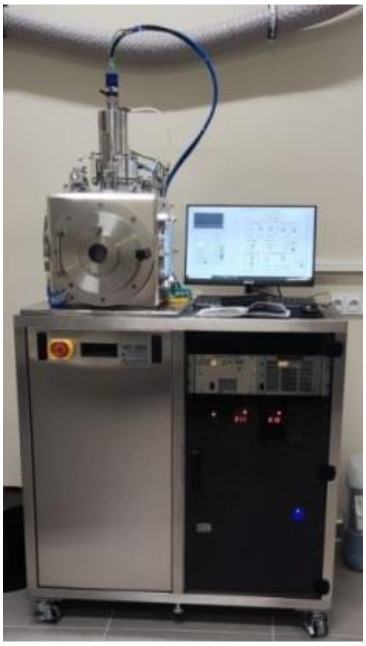

**Figure 1** Nanomaster coating device NPE-4000

The AlNiCo alloy samples were washed in an ultrasonic cleaner and then put into the device chamber, where they were cleaned in plasma in a vacuum of approx. $3.5 \cdot 10^{-5}$ Torr. After that, Al and Ag coatings were deposited using an aluminum or silver target, respectively. **Table 1** shows the coating application parameters. Where DC is the direct current in watts and Ar is the flow rate in standard cubic centimeters per minute (sccm).

**Table 1** Al and Ag coating application parameters

| Element | Ar, sscm | DC, W | Time, min | Thikness of layers, µm |
|---|---|---|---|---|
| Al | 30 | 20 | 90 | 0.62 |
| Al | 30 | 20 | 135 | 0.98 |
| Al | 30 | 20 | 180 | 1.87 |
| Al | 30 | 20 | 240 | 5.94 |
| Ag | 30 | 20 | 360 | 0.56 |

The chemical composition of the obtained coatings was analyzed using a JEOL 7100F scanning microscope with field emission. The microscope is equipped with an OXFORD Instruments X-max EDS detector and a BSE detector. Magnification range: from 10 to 1 000 000 times. Resolution: 1.2 nm at 30 kV, 3 nm at 1 kV. Range of accelerating voltage changes: from 100 V to 30 kV Sample dimensions: maximum diameter 40 mm, maximum height 25 mm. The tests were carried out with the following parameters:

- Acceleration Voltage 15kV,
- sampling current (Probe Current) 1 nA,
- Work Distance 10 mm.

The AZtec program was used to analyze the obtained data. First, a linear analysis was performed to confirm the presence of the applied layer on all samples. **Figure 2** shows a photo of the area subjected to linear analysis for the Al deposition time of 135 minutes. **Figure 3** shows distribution of elements along the designated line for 135 minutes deposited of aluminium.

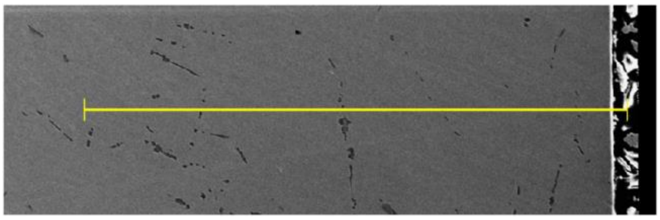

**Figure 2** Photo of the area given by linear analysis on a scanning microscope for an Al deposition sample, t=135 min

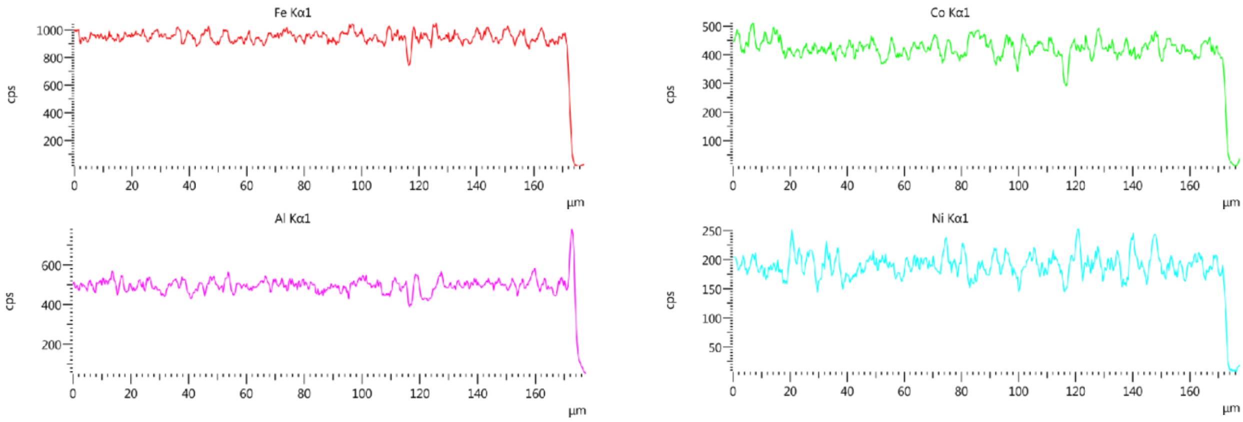


**Figure 3** Distribution of elements along the designated line for the Al deposited sample, t=135 min

The samples were analyzed for the thickness measurements of the deposited layers. **Figure 4** shows examples of coating thickness measurements for a sample with an aluminum coating applied for 135 minutes.

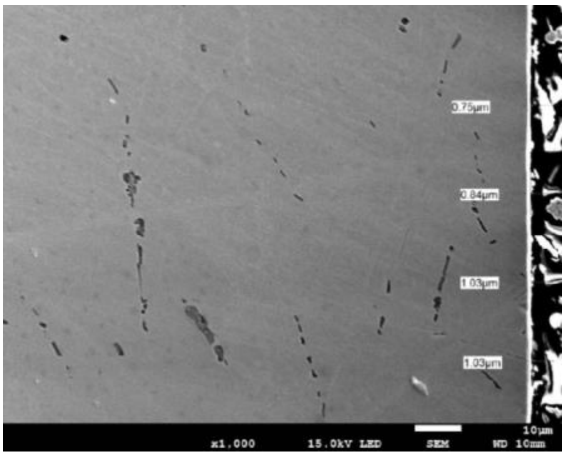


**Figure 4** Coating thickness measurements

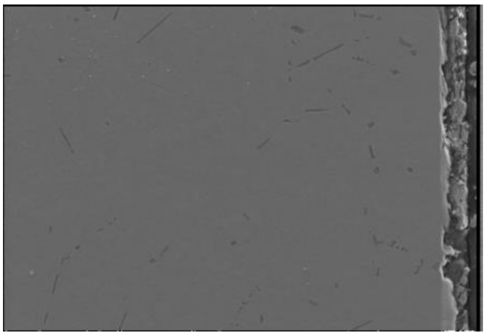

**Figure 5** Photo of the area mapped on a scanning microscope for a sample of aluminum coatings applied during 240 min

In addition, mapping of elements was performed in order to check whether the deposited layer is actually reflected in the obtained maps of chemical composition. Sample maps are shown in **Figure 5** and **Figure 6** for a 240 minutescoating of the applied aluminum.

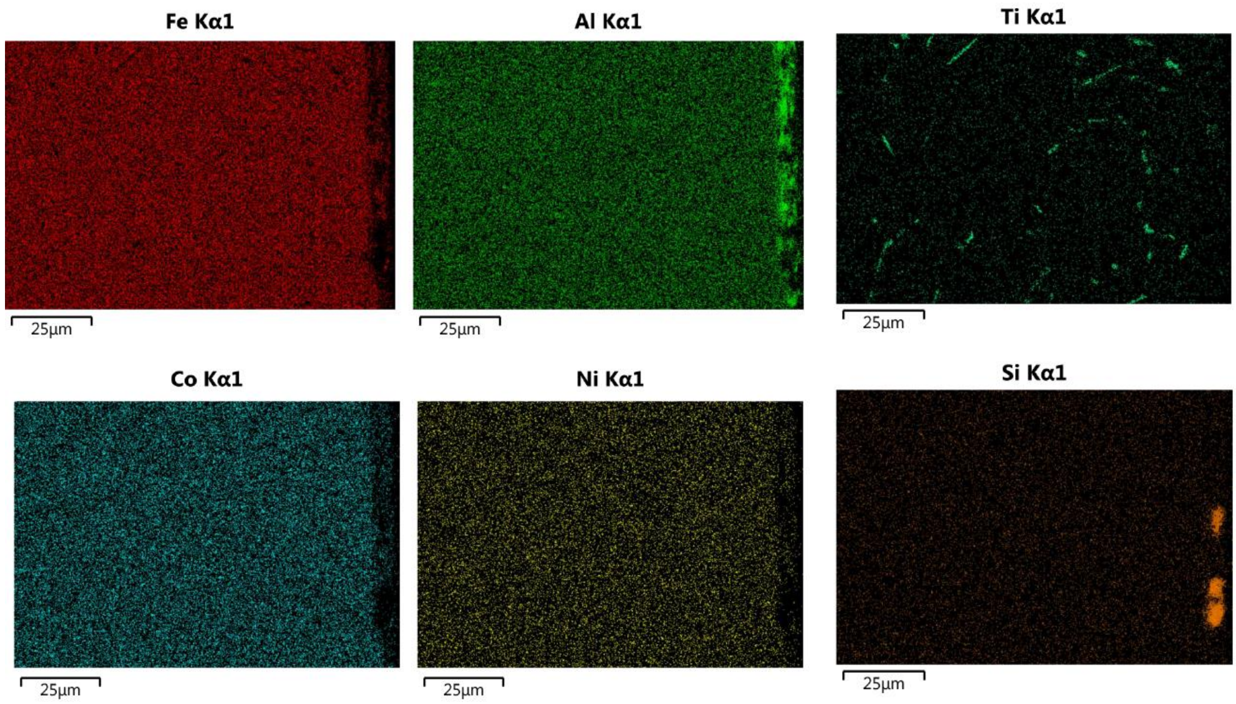


**Figure 6** Distribution of elements in the analyzed area for a sample with an Al coating applied for 240 min

## 3. CONCLUSION

Distribution of elements in the analyzed area for a sample with an Al coating for 240 min The purpose of the coatings is to improve selected properties, including mechanical, tribological, corrosion and bactericidal properties. The parameters of the deposition process should be selected so that the finished item meets the intended features and functions. The most important factor is the choice of the right coating - the choice of material to be applied. During the coating application process, it is important to properly prepare the substrate, as well as to select the parameters of the application process: power, time, amount of gas flow.

- The performed analysis of the distribution of chemical elements along the line clearly indicates the presence of aluminum and silver layers. The obtained results clearly indicate different sizes and widths of the peaks for aluminum, which is caused by different layer thicknesses for a given sample,
- Mapping of the surface of the samples showed that the elements are evenly distributed. In the case of Al deposition samples, the deposited layer is less visible on the maps for 90 minutes, which is caused by small coating thicknesses in these samples,
- It has been observed that silver coatings take much longer to apply the same thickness of coating as aluminum coatings,
- Aluminum (Al) coatings deposited in 135 minutes had a thickness of about 1 μm
- Silver (Ag) coatings were applied within 5 hours and reached a thickness of about 0.556 μm. After examining the chemical composition using a scanning electron microscope, it was noted that this coating consisted mainly of silver, as well as elements such as magnesium, aluminum, silicon, iron and cobalt, which originated from the substrate.

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