

Jerzy Bieliński^{1*}, Anna Sałacińska², Rafał Kozera², Alicja Bielińska¹, Anna Boczkowska²
Krzysztof J. Kurzydłowski²

¹ Politechnika Warszawska, Wydział Chemiczny, ul. Noakowskiego 3, 00-664 Warszawa, Poland

² Politechnika Warszawska, Wydział Inżynierii Materiałowej, ul. Wołoska 141, 02-507 Warszawa, Poland

* Corresponding author. E-mail: bielin@ch.pw.edu.pl

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CATALYTIC ACTIVATION OF CARBON FIBRES IN ELECTROLESS PROCESS OF FABRICATION OF METALLIZED CARBON FABRICS

The influence of metallization process parameters such as bath composition and deposition rate on the thickness and morphology of coatings were investigated. The role of bath composition (Ni-salt, reducer, complexing-buffering additive, stabilizer, surfactants), pH value, temperature and deposition time were studied. The requirements for C-fiber/Ni-P/Al alloy composite fabrication are limited to the P-content to 2÷3 wt.% and coating thickness to less than 1 µm. The metallization process, after proper carbon fiber pretreatment, was performed in baths containing: NiSO₄, NaH₂PO₂, glycine, stabilizers and wetting agents. The pH value was shifted to the range of 4.5÷8.5, temperature 60÷80°C and deposition time from 5 to 60 min. As a substrate for the metallization process, roving and fabrics manufactured by Tenax (3-24k filaments in bundle with 7 µm diameter), as well as mullite test samples were used. The results of the experiments, limited to changes of pH in the bath, indicated that the ratio of NiSO₄/NaH₂PO₂ concentration is the main factor determining the P-content and Ni-P deposition time. It is the best factor for fixing the coating thickness. The increase of the Ni-P coating thickness rate also depends on the quantity of carbon fibers in the roving.

Keywords: electroless Ni-P deposition, carbon fibers, catalytic activation, ultrasounds, microstructure

AKTYWACJA KATALITYCZNA WŁÓKIEN WĘGLOWYCH W PROCESIE BEZPRĄDOWEGO WYTWARZANIA METALIZOWANYCH TKANIN WĘGLOWYCH

Przedstawiono badania nad bezprądową metalizacją włókien węglowych poprzez osadzanie na ich powierzchni warstw Ni-P. Celem pracy było szczegółowe określenie roli parametrów decydujących o morfologii i grubości powłok Ni-P, takich jak: składu roztworu (stężenia soli niklu i reduktora, związku buforującego, stabilizatora, zwilżacza) i jego pH oraz temperatury, a także czasu osadzania i ilości oraz rodzaju metalizowanych włókien. Ze względu na wymagania procesu infiltracji pometalizowanych tkanin węglowych przez stopy aluminium korzystne jest ograniczenie do minimum 2-3% mas. zawartości fosforu w powłokach Ni-P oraz grubość tych powłok poniżej 1 µm. Proces metalizacji, po odpowiednim przygotowaniu włókien, prowadzono z roztworów zawierających NiSO₄ - 0,1/0,2 M; NaH₂PO₂ - 0,1/0,2 M; glicynę - 0,2/0,42 M oraz stabilizatory - NaNO₂, NaAsO₂, NH₄SCN, Na₂MoO₄, SC(NH₂)₂ (stężenia 0,01÷1 mM) i środki powierzchniowo czynne (sole amin czwartorzędowych o stężeniu 0,5÷1 mM). Wartość pH zmieniano od 4,5 do 8,5, a temperaturę od 60 do 80°C przy czasach osadzania od 5 do 60 minut. Podłożem były zarówno wiązki włókien węglowych firmy Tenax, zawierające od 3k do 24k włókien o średnicy 7 µm, jak również tkaniny z tych wiązek oraz kształtki mullitowe (jako podłoża testowe). Stwierdzono, że w warunkach ograniczonych zmian pH podstawowym parametrem kształtowania składu warstw Ni-P jest stosunek stężeń NiSO₄/NaH₂PO₂ w roztworze, a grubość powłok najlepiej jest ustalać poprzez zmiany czasu metalizacji. Szybkość przyrostu grubości powłoki Ni-P zależy też istotnie o rodzaju wiązek.

Słowa kluczowe: bezprądowe osadzanie Ni-P, włókna węglowe, katalityczna aktywacja, ultradźwięki, mikrostruktura

INTRODUCTION

The application of MMC composites reinforced with carbon fibres allows a significant decrease in the density of the material and its thermal expansion. On the other hand such a composite exhibits improved strength and other functional properties. These advantages lead to a wide possibility of applications of MMC in the industry [1-5]. However, a barrier for the technology and final products is the insufficient wettability of car-

bon fibres with a liquid metal e.g. of aluminium alloys. Additionally the formation of harmful carbides on the carbon fibre/aluminum alloy interface is a significant problem [1, 2, 4]. A solution to this issue can be the fabrication of special layers on the surface of the carbon fibres to prevent or at least decrease to a minimum the contact of liquid metal with the fibres [1, 2, 4]. In literature many examples of such layers fabricated by

different methods e.g. PVD, CVD, sol-gel and chemical electro and electroless deposition can be found [1, 2, 4, 7, 8]. Selection of the proper method, advantages and disadvantages of using them in specified applications are widely described in publications including our previously works about electroless catalytic metal plating of carbon fibres [2, 4, 8, 9-17]. The electroless-catalytic method of the metallization of carbon fibres is distinguished among others in the possibility to fabricate uniform coatings on complicated profiles and shaped elements. Preponderance of this method is the result of undirected transport of reagents, which is absent in other methods with e.g. an electric field. That is why the processes of nickel or copper plating are still under development for different applications in MMC [4, 9-17].

In electroless catalytic metallization, an additional step namely the deposition of catalyst grains is required. Catalyst grains allow the initiation of a further deposition process which is autocatalytic [18]. Even superficial literature review indicates that in most publications, the parameters of the deposition of catalyst grains are similar to those suggested in the most commonly known electroplating monographs [19-21]. This paper is focused on the development of the parameters of this stage, intended specially for carbon fibre and fabric metallization.

EXPERIMENTAL PROCEDURE

The carbon fibres used in this work were Tenax HTA40, roving in bundles containing 3k or 24k fibres with a diameter of 7 μm . The bundles were cut into 10-20 cm sections in length. The protective commercial epoxy layer deposited on the fibres was removed by annealing in an oven for 1 h at 450°C in air atmosphere [22]. The next step of fibre preparation before the metallization process was the surface catalytic activation. The process consists of the following operations: first, bundles were immersed for 2-15 min in a sensitization solution based on Sn(II) salt, next in a catalyst solution based on Pd(II) salt. At the end of each process rinsing in distilled water was performed. The addition of organic wetting compounds (surfactants) was also applied together with the tested solutions. In the previous tests with aluminum oxide powders, an advantageous influence of the reactivation of a Pd catalyst [23],- through to the applying of a pre-reduction solution, i.e. sodium dioxophosphate(I) (Na-hypophosphite), on the bases prior to the subsequent metallization process, was found. A similar reduction pre-treatment condition with a solution of 0,2M NaH_2PO_2 + 10 mM NiSO_4 , 70°C in this work was applied.

After the catalyst deposition, the samples were moved to thermostatic containers (100-200 cm^3) with a Ni-P metallization bath. The vertically fixed carbon fibre roving samples were immersed in an electroless solution. The operation conditions were as follows:

bath temperature of 70°C and deposition time of 15-30 min. The composition of the metal plating solution included: nickel sulphate (II) NiSO_4 - 0.1 M; sodium dioxophosphate(I) (NaH_2PO_2 , reductant) - 0.2 M; aminoacetic acid (glycine, buffering and complexing agent) - 0.2-0.21 M. The pH value was set at 8.5. In most cases, before the deposition process, a bath stabilizer- thiourea $\text{SC}(\text{NH}_2)_2$ (TM; 0,02 mM) and surfactant (wetting agent) - cetyltrimetyloammonium bromide (CTAB; 1 mM) were added to the solution.

The chemical composition and thickness of the Ni-P coatings were determined [24]. The rapidity of the deposition process was determined as the mass of deposited Ni on a specified length of bundle. The second parameter was the k_m value described as the relation between the mass of Ni-P coatings to the mass of the carbon fibres (m_{NiP}/m_C). The morphology of the carbon fibre surface with Pd grains as well as the morphology of the Ni-P coatings were observed using SEM (HRSEM HITACHI S5500).

RESULTS AND DISCUSSION

The aim of the performed studies was to determine the role of the selected parameters of the catalytic activation processes on the carbon fibre surface.

Compositions of Sn(II) and Pd(II) solutions

The majority of solutions used for the sensitization and activation of various non-metallic substrates are based upon SnCl_2 and PdCl_2 with an addition of HCl. A review of monographs and handbooks shows a wide concentration range of these salts in the solutions - between 10 and 100g/l for SnCl_2 , and between 0.1 and 2 g/l for PdCl_2 [19-21, 25-27]. There are two methods for applying the mentioned solutions. The two-step catalytic activation consists of sensitization and activation processes: subsequent immersion of the samples in Sn(II)- and Pd(II)-salt solutions. In the one-step catalytic activation, the samples are immersed in a colloidal solution of a mixed system Sn(II)-Pd(II)-salt, followed by an additional post-activation (acceleration) [25-27]. The mechanism of Pd-catalyst deposition is very complex and usually described by the simplified equation:



It is essential that reaction (1) proceeds on the substrate surface covered during sensitization with a layer of partly hydrolysed and oxidized tin(II) and tin(IV) compounds (for instance $\text{Sn}(\text{OH})\text{Cl}$, H_2SnO_3) being sparingly water-soluble. That gives a hydrophilic nature to the substrate surface. The analysis of publications concerning the metallization of carbon fibres has shown an unavailability of studies on the stage of preparation for electroless plating. Data on spreading the Pd catalyst is also incomplete (Table 1). The changes in SnCl_2 and PdCl_2 concentrations are only confined to the 10-20 g/l

and 0.1÷1 g/l range. The process duration at room temperature was between 1 and 15 min. Using ultrasounds for improved solution penetration between the fibres has been also mentioned.

TABLE 1. Literature data on CF pretreatment sequence

TABELA 1. Sposoby aktywacji włókien węglowych - dane literaturowe

Type of substrate/coating	Pretreatment sequence	Sensitization/activation	Ref.
Fortfil 04T - Fortral Ni-P	- sensitization - activation	10 g/l SnCl ₂ + 40 ml/l HCl 0.2 g/l PdCl ₂ + 10 drops /l HCl	[10]
PAN - fabric Indie Ni-P	- desizing acetone+MeOH - sensitization - activation	SnCl ₂ ok. 10 g/l; 1 min PdCl ₂ ok. 1 g/l; 5 min	[11]
AS4 Hexcel Composite. Ni-P	- heat treatment - sensitization - activation + UD*)	40 ml/l HCl + 20 g/l SnCl ₂ ·2H ₂ O; 15 min 0.2 g/l PdCl ₂ + 2.5 ml/l HCl	[12]
AS-4 Hexel Cu	- heat treatment 500°C - sensitization - activation	SnCl ₂ ·H ₂ O 12 g/l +HCl 40 ml/l; 15 min PdCl ₂ 0.2 g/l + HCl 2.5 ml/l; 15 m, UD	[13]
PAN Mitsubishi Ni-P	- desizing 450°C - etching? CrO ₃ + H ₂ SO ₄ - sensitization - activation	SnCl ₂ 20 g/l +HCl 40 ml/l, 0,25 g/l PdCl ₂ +2.5 ml HCl, UD	[14]
T300C T700SC Toray Ni-P	- cleaning - sensitization + UD - activation + UD	20 g/l SnCl ₂ ·2H ₂ O + 40 ml/l HCl 0.25 g/l PdCl ₂ + 2.5 ml/l HCl	[28]
AS4 Hexcel Composites Ni-P	-cleaning -drying 110°C -sensitization -activation + UD	40 ml/l HCl + 12 g/l SnCl ₂ ·2H ₂ O; 15 min 0.2 g/l PdCl ₂ + 2.5 ml/l HCl,UD	[29]
PAN Formosa Co. Ni-P	-cleaning -sensitization + UD -activation + UD	40 ml/l HCl + 20 g/l SnCl ₂ ·2H ₂ O 0.25 g/l PdCl ₂ + 2.5 ml/l HCl, UD	[30]

Thus, for the present study, the precondition was determined to use the sensitization and activation sequence for the aluminium oxide powder metallization [23] material featured by an equally high surface development. For example, the surface of a 1 g Alcoa A-16 aluminium oxide sample is about 8 m². The geometrical surface of a 24k, 10 cm long fibre bundle is about 0.5 m², the size of a 10x10 cm fabric patch amounts to more than 100 m². It was also assumed that the activation solution will be the major study object, since the concentration changes in PdCl₂ are more important in the Pd catalyst deposition process than the concentration of SnCl₂ [25, 27]. The following conditions for experiments with the 3k, 8 cm long fibre bundles were applied:

- sensitization of SnCl₂ 10 g/dm³ + HCl (37%) 20 cm³/dm³; time - 15 min,

- rinsing in distilled water - 2 min,
- activation of PdCl₂ (between 0.01 and 1 g/l) - 5 min.
- rinsing in distilled water - 15 min.

The samples with the deposited Pd-catalyst were then metallized for 15 min. Since rinsing the samples consisted in submersion in distilled water, without any additional stirring or motion, self-decomposition of the metal plating solution occurred. The results of this experiment series are presented in Table 2. It was found that maximum coverage on the fibres was obtained at 1g/l PdCl₂ (HCl 2 ml/l, pH = 1.6), as a result under complete penetration by palladium salt between the fibres. At the same time, the remnants of the catalyst salt were not completely rinsed out with water, and that was the cause for self-decomposition of the metal plating solution. The conclusions applicable for further studies on the metallization process of carbon fibre bundles were that the capacity of the rinsing water container (from the present 100 ml up to 300 ml) has to be enlarged, and an exchange of water provided while post-activation rinsing is carried out. The application of a magnetic stirrer during water rinsing enhanced the motion intensity of the liquid surrounding the filaments. However, the original operation time-sequence was retained.

TABLE 2. CF metallization at different PdCl₂ concentrations

TABELA 2. Metalizacja włókien węglowych dla różnych stężeń PdCl₂

Concentration of PdCl ₂ g/dm ³	Plating time (min) before self-decomposition	Deposition Ni mass mg	Deposition rate mg/cm ² ·min
1	2	14	0.88
0,5	10	68	0.85
0,2	14	87	0.72
0,1	without SR*	54	0.45
0,05	without	SR	12
0,10,02	without SR	0	0
0,01	without SR	0	0

*) SR – decomposition of electroless bath

Certain studies on the Pd activation process [25, 27] emphasise that the pH value of the PdCl₂ solution should be optimised for various substrates. Thus, a series of activation tests in solutions with varying pH values which were subsequently changed to a 1.1-1.6-2.1-2.6 pH sequence (by addition of HCl) were performed. It was found that the best coating of the fibre bundle with Ni-P can be obtained after the activation process in PdCl₂ solutions at pH = 1.6÷2.1. The solution with pH = 1.6 is recognised as a standard one for further studies on carbon fibre metallization.

Modification of solutions and catalyst spreading method

In order to improve the penetration of the solution into the bundles, surfactant agents were added [25]. Anionic (sodium dodecylsulfate) and cationic

(tetramethylammonium iodide) wetting agents with a $1\div 10$ mM concentration were subsequently introduced into standard solutions of Sn(II) and then Pd(II). Enhanced inflow of the modified solutions should result in a better coating of the fibre surface with Pd grains, and this in turn, has to cause a higher coverage of the fibres by the Ni-P deposit (at the same metal plating time). The metallization process was carried out under standard conditions described in methodology, with a solution containing additives of stabilising and wetting agents. The results of the test series proved that the surfactant additives in $\text{SnCl}_2/\text{PdCl}_2$ solutions caused no essential change to the coating rate of the fibres by the Ni-P layer; between 3.6 and 4.2 mgNi/mgC, for the metallization of 6k fibres (and $3.1\div 3.8$ for deposition after activation in solutions without surfactant additives). To compare, the deposition rate ranged between 21.3 and 24.8 $\text{mg/m}^2\cdot\text{s}$ on mullite forms (which provide for very good penetration by solutions into the substrate) was applied. The wetting agent also facilitates removal of hydrogen bubbles formed during the electroless reaction of Ni-P deposition [15, 16, 18].

The analysis of the literature data (Table 1) indicates that the use of ultrasounds could be effective. Depending on the capacity of the generator, ultrasounds might cause the vibration of fibres, or lead to cavitation effects (thus causing easier degassing of the fibre surrounding space). However, publications have not provided enough quantitative data and conclusions [31, 32]. Preliminary studies were carried out to determine the optimum use of ultrasounds generated in laboratory washers/rinsers: UD2 - 200 W and UD3 - 1200 W (IS20 type manufactured by Intersonic). The reactivation (pre-reduction) and deposition processes of Ni-P on the bundles were carried out in the latter rinsers designated for the metallization of the carbon fibre fabric samples. Table 3 shows the processing sequences of the fibre filament treatment through the subsequent operations, from sensitization up to metal plating.

TABLE 3. Deposition of Ni-P for different carbon fiber treatment

TABELA 3. Wyniki osadzania włókien węglowych po zastosowaniu różnych wariantów aktywacji

No.	Sn(II) min	Rinse after Sn min	Pd(II) min	Rinse after Pd min	RE* min PF**	Ni-P plating min	$m_{\text{Ni}}/m_{\text{C}}$ mg/mg
1	15	2	5	15	-	15	4.61
2	15	2	5	15	-	15UD	3.64
3	15	2	5	15UD	-	15	5.30
4	15	2	5UD	15	-	15	4.95
5	15	2	5	15	5	15	5.11
6	15	2	5	15	5UD	15	4.80
7	15	2	5	15	5UD	15UD	3.37
8	15	2	5UD	15UD	5UD	15UD	4.75
9	15	2UD	5UD	15UD	5UD	15UD	2.51
10	15UD	2UD	5UD	15UD	5UD	15UD	2.56

*) RE - reactivation

**) PF - Na-hypophosphite 0,2

The results indicate the complex nature of the ultrasonic effects on the course of both the partial processes and the final one. The application of ultrasounds only in the metallization step might cause a drop by even 20% in the amount of the Ni-P quantity deposited. An enhancement of the solution penetration between the fibres caused by the ultrasonic action provides an increase in the coating thickness. The performed experiments indicate that ultrasonic action also has adverse effects on the process run. This relates to the mechanical removal of a part of the Pd catalyst grains during activation, post-activation rinsing and the Ni-P deposition processes. At the same time, the removed Pd grains are deposited at the bottom of the metallization container and sometime initiated self-decomposition of the solution. The catalyst reactivation (pre-reduction) operation could be a good solution to prevent this phenomena [2, 3, 33].

The substrate after the activation of Pd and their preliminary water-rinsing, are briefly immersed in a reductant (Na-hypophosphite) solution, and then transferred directly to the metallization bath. The characteristic structures of the carbon fibre surface for various Pd grain production variants are presented in Figure 1. For standard preparation of the catalyst (without using ultrasounds and reactivation) a dense arrangement of Pd grains is typical and the grains easily undergo the removal process (Fig. 1a,b). The application of ultrasounds causes the removal of large grains. Fine grains are visible that are more firmly bound to the fibre surface (Fig. 1c,d). rinsing the fibres in an Na-hypophosphite solution (reactivation) provides the attainment of compact, fine-grain Pd areas (Fig. 1e,f).

The nature of the catalyst grains, i.e. metallic Pd, as produced in the present study under a 2-stage activation process, is a fine-crystalline. This feature reduces their adhesion on the carbon fibres. The Ni-P layers deposited on the Pd grains are characterized by a lower degree of crystallinity and the Ni-P hemispheres are better bonded to functional groups of the substrate surface, i.e. carbon fibers. The Ni(II)-containing complexes of organic hydroxy-acids are more persistent than the Pd(II)-containing ones [34]. The above observation led to further modification of the preparatory sequences of the catalyst for Ni-P deposition. A low-concentration ($1\div 10$ mM) Ni(II) salt was introduced into the Na-hypophosphite solution for reactivation. Reactivation after rinsing leads to coating the crystalline catalyst grains with nano-size domes - Ni-P nano-layers. Such structures again expand into dense areas with Pd/Ni-P grains (Fig. 1g). Submersion of the samples in the solution for electroless nickel-plating, together with the catalyst produced, leads to the final structures of the Ni-P layers featured by relatively large hemisphere grains. The Pd/Ni-P catalyst is much more resistant to ultrasonic impact.

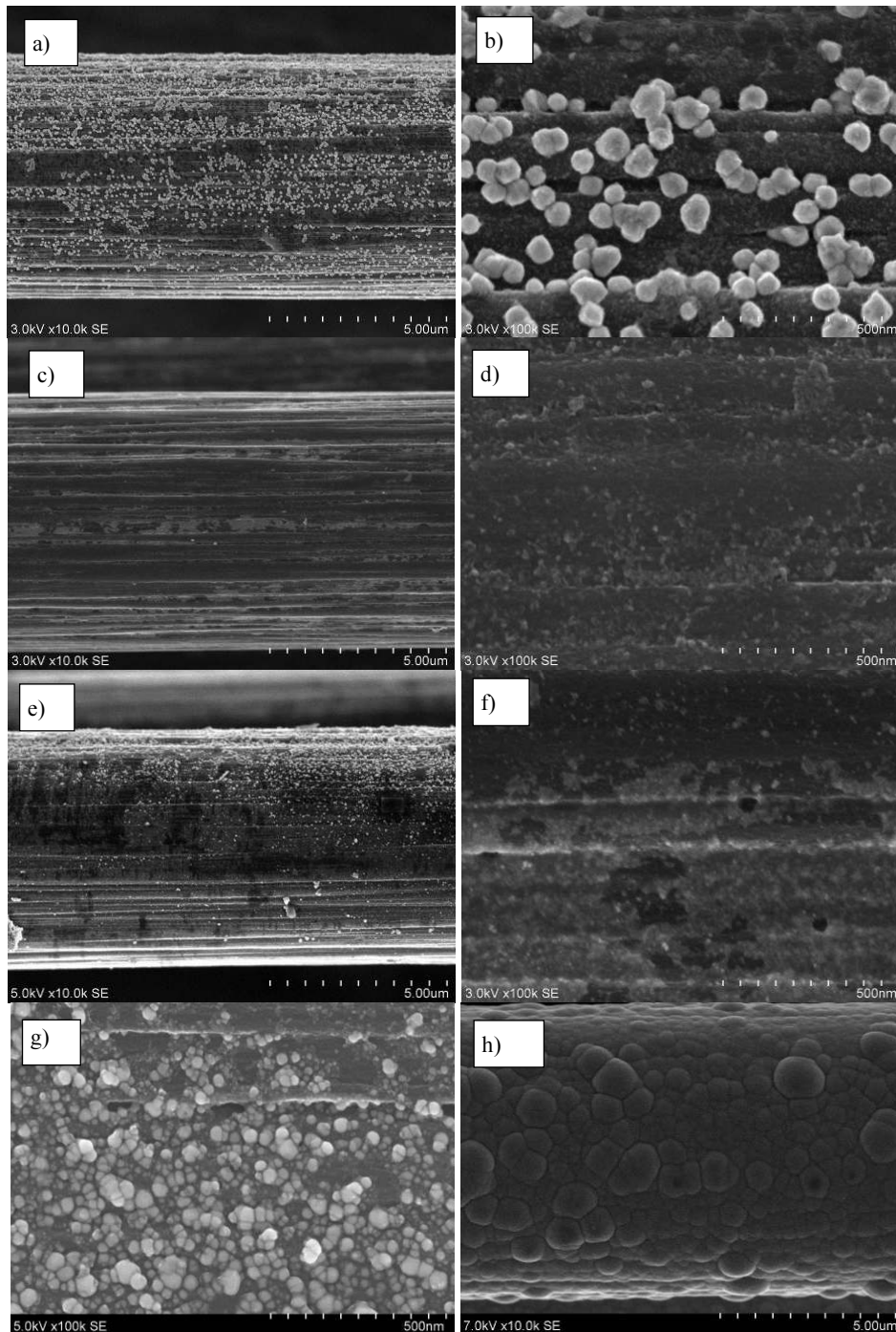


Fig. 1. Typical structures of Pd, Pd/Ni-P catalyst and Ni-P layer: 1a,b) after standard Sn/Pd procedure; c,d) after additional UD rinse; e,f) UD rinse after Pd reactivation, g) UD rinse in reactivation bath with Ni(II); h) final structure of Ni-P layer on CF

Rys. 1. Typowe struktury powierzchni włókien po aktywacji Pd i z warstwą Ni-P: 1a,b) po standardowej procedurze aktywacji Sn/Pd; c,d) po płukaniu w UD; e,f) płukanie w UD po aktywacji Pd; g) zastosowanie UD podczas reaktywacji w roztworze zawierającym Ni (II); h) struktura warstwy Ni-P na włóknach

CONCLUSIONS

The tests and observations indicate that:

- The same sequence of Pd deposition as for ceramic powders can be used for the metallization of carbon fibres.
- The most difficult issue in catalyst deposition on carbon fibres consists in providing an appropriate penetration of solutions between carbon fibres in the

bundles, such as in the case of fabrics. The addition of wetting agents to the metallization solution did not bring positive results, while the application of ultrasounds proved to be very useful.

- In spite of using stabilizers in the metallization bath for bundles of fibres (or, even to a greater degree, fabrics), it often leads to self-decomposition of the metallization bath, unacceptable from the technological viewpoint. In order to avoid this effect it is necessary

to very carefully remove all the residuals of Pd(II) salt from the inter fibre space together with the loosely embedded large Pd grains. The application of ultrasounds and an additional process of reactivation (pre-reduction) before metallization using a solution of reducer is helpful in this process.

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