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Synthesis and Characterization of Fe-W and Ni-W Composite Coatings

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Abstract. This paper reports some results concerning the preparation and magnetic properties of Fe-W and Ni-W composite coatings obtained by co-deposition process. Fe-W and Ni-W composite coatings were electrochemically synthesized from iron and nickel sulphate baths, respectively, containing W nanoparticles with sizes between 500 and 700 nm.

Key words: co-deposition process, composite coatings, magnetic properties.

1. Introduction

Electroplated composite materials can be synthesised by co-deposition process of a metal and a powder suspended in electrolyte [1]. The embedded particles can be selected to fulfil specific mechanical, electrical, piezoelectric or magnetic properties in thin coatings. Inclusion of tungsten in Ni deposits affects the deposition rate, composition and deposit properties such as hardness, thermal stability, wear, corrosion and electrical resistance [2, 3].

Three main mechanisms [4] were previously suggested to explain the difference in the ability to deposit various types of solid particles:

(1) Mechanical entrapment: the particles are driven to the cathode by vigorous bath agitation and are incorporated in the growing metal layer only if the contact period is long enough and the metal deposition rate is high enough;

(2) Electrophoresis: charged particles are moving under the influence of an applied electric field. The electrophoretic velocity is directly proportional to the zeta potential (ξ-potential), which is defined as the potential at the shear surface;

(3) Adsorption: near the cathode, particles will be subjected to various attractive forces. Once adsorbed onto the cathode, the particles are embedded in the growing metal layer.

In this work, we report some results on the preparation and magnetic properties of Fe-W and Ni-W composite coatings obtained by co-deposition process.

2. Experimental

Composite Fe-W and Ni-W coatings were electrochemically synthesized from iron and nickel sulphate baths, respectively, containing commercial W nanoparticles (from Merck).

Electrochemical deposition of the samples was performed using an electrochemical cell with three electrodes. The synthesis of all electrodeposits containing W nanoparticles was controlled by a potentiostat VOLTALAB PGZ 100.

Figure 1 shows schematically the electrodeposition procedure for the preparation of Fe-W and Ni-W composite coatings.

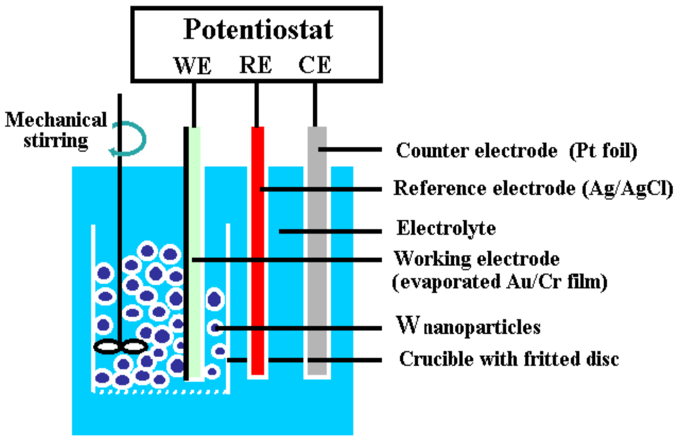

Fig. 1. Schematic diagram of the electrodeposition process.

The composite coatings were electrodeposited onto non-magnetic substrates. We have used glass substrates with a surface area of 2 cm^2, over which an electroconductive thin film (Au/Cr) of about 65 nm was deposited by thermal evaporation.

For the electrodeposition of composite coatings a platinum piece was used as counter electrode and an Ag/AgCl electrode as reference electrode.

The process was carried out with intensive mechanical stirring (1700 rpm) to maintain the W nanoparticles in suspension.

The size distribution of the W nanoparticles was determined by Dynamic Light Scattering (DLS), with a Nanotrac device. W nanoparticles are ranging between 500 and 700 nm. In Figure 2 is presented the size distribution of W nanoparticles.

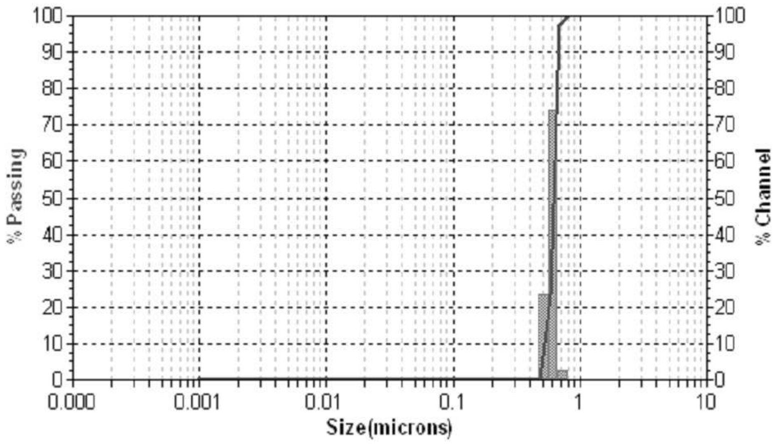

Fig. 2. Size distribution of W nanoparticles.

The plating bath for the electrodeposition of Fe-W and Ni-W composite coatings consisted of $FeSO_4{\cdot}7H_2O$ (420 g/l), K_2SO_4 (1 g/l), $H_2C_2O_4{\cdot}2H_2O$ (1 g/l) and $Al_2(SO_4)_3{\cdot}18H_2O$ (85 g/l), respectively $NiSO_4{\cdot}7H_2O$ (300 g/l), $NiCl_2{\cdot}6H_2O$ (40 g/l) and H_3BO_3 (30 g/l). The plating conditions use to obtain Fe-W and Ni-W composite coatings are shown in Table 1.

Table 1. The plating conditions used
to obtain Fe-W and Ni-W composite coatings

Plating conditions	**Composite coatings**	
	Fe-W onto Au/Cr thin film	**Ni-W onto Au/Cr thin film**
Temperature (°C)	50	50
pH_{bath}	1.5	4
Current density (mA/cm^2)	100–150	10–20
Voltage (V)	6–8	2–3
Content of W nanoparticles in solution (g/l)	20–40	20–40
Electroplating time (min)	10	15

The crystallographic structure of electroplated Fe-W and Ni-W composite coatings was examined by X-ray diffraction (XRD), using monochromatized Co-Kα radiation ($\lambda = 0.1789$ nm).

The magnetic hysteresis loops as well as the temperature dependence of the coating layers magnetization were measured using a vibrating sample magnetometer (VSM), in a maximum external magnetic field of 600 kA/m.

3. Results and discussion

3.1. Morphology of the electroplated composite coatings

In Figs. 3 and 4 are shown the morphology of the surface and cross-section of Fe-W and respectively Ni-W composite coatings electrodeposited onto Au/Cr thin film. The images clearly show the presence of W nanoparticles embedded into the Fe and respectively Ni thin electrodeposited layers.

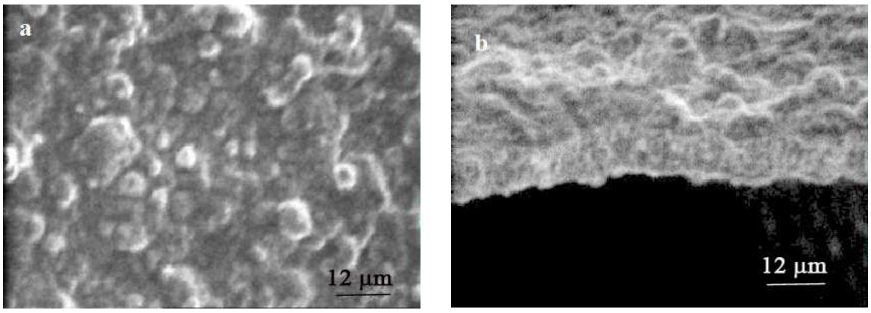

Fig. 3. SEM micrographs of Fe-W composite coatings: (a) surface morphology; (b) cross-section.

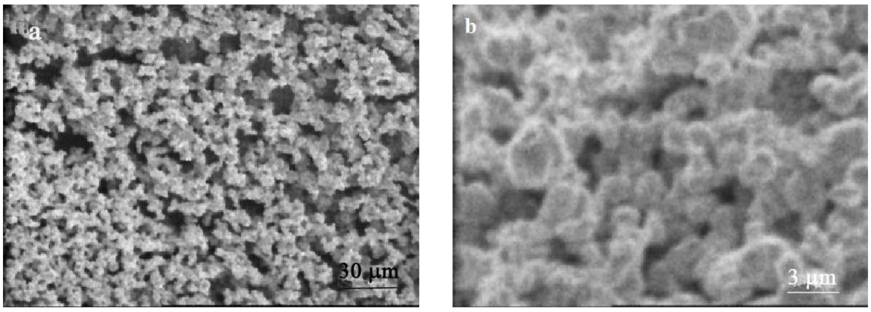

Fig. 4. SEM micrographs of the surface of Ni-W composite coatings.

During vigorous stirring of the plating bath solution, W nanoparticles are driven to the cathode and are embedded into the growing Fe or Ni layers. Fe-W and Ni-W composite coatings present a matt and rough surface, the roughness of obtained composites being affected by amount of embedded W nanoparticles.

The thickness of the obtained Fe-W composite coatings onto Au/Cr thin film is ranging between 3–12 μm, whilst the one of Ni-W composite coatings is between 2.5 and 6 μm.

Fe-W and Ni-W electrodeposited composite coatings show good adhesion to the Au/Cr thin film. The W nanoparticles are uniformly embedded into the iron and nickel matrix with tendency to agglomerate.

3.2. Structural features of the composite coatings

The X-ray diffraction patterns of Fe-W (Fig. 5) and Ni-W (Fig. 6) composite coatings onto Au/Cr thin films show the presence of sharp reflexes characteristic to Fe or Ni matrix and the embedded W nanoparticles, but also the sharp peaks corresponding to Au/Cr thin film substrates.

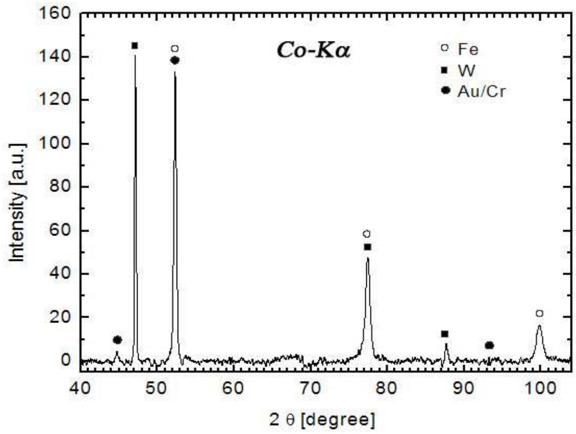

Fig. 5. X-ray diffraction pattern of Fe-W composite coating.

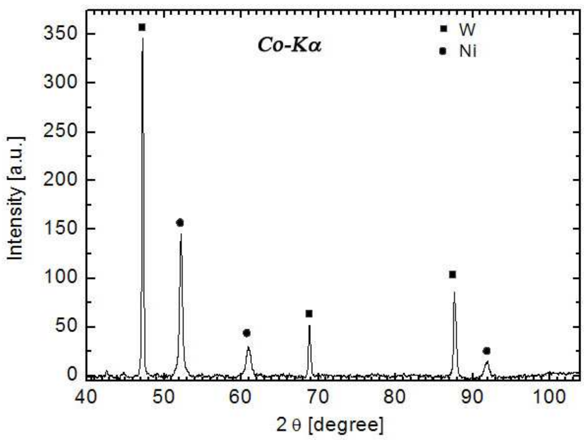

Fig. 6. X-ray diffraction patterns of Ni-W composite coating.

3.3. Magnetic properties of Fe-W and Ni-W composite coatings

In Fig. 7 are presented the thermomagnetic curves (a) and the magnetic hysteresis loops (b) as a function of the W content in plating bath for Fe-W composite coatings electrodeposited onto Au/Cr thin film.

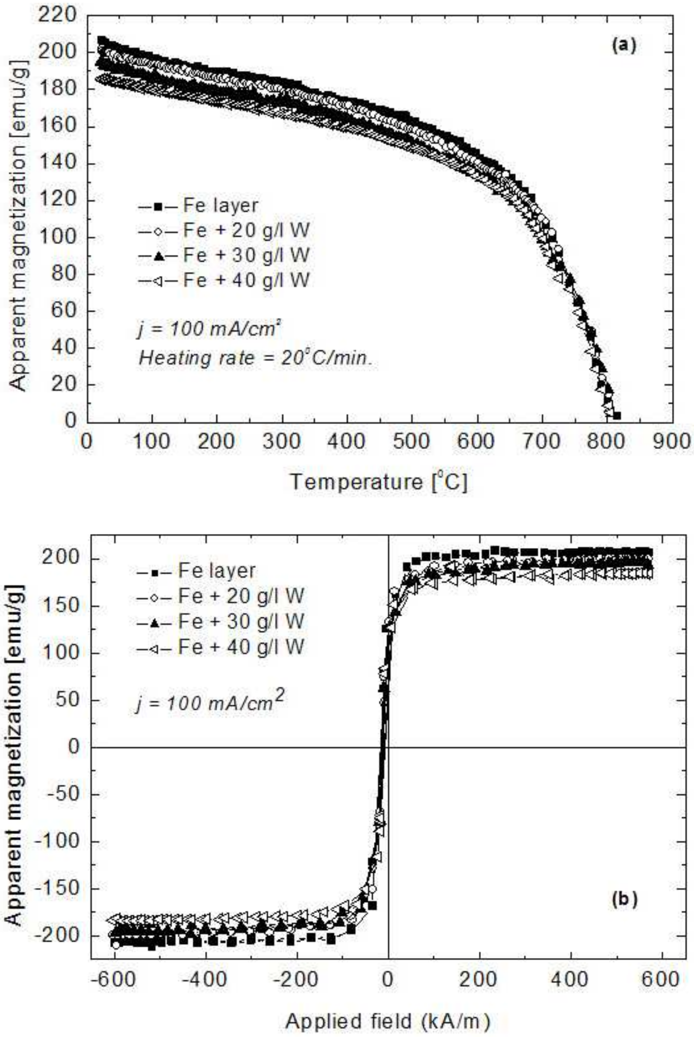

Fig. 7. Temperature dependence of the magnetization and magnetic hysteresis loops as a function of the content of W nanoparticles in the plating bath.

Figure 8 presents the thermomagnetic curves (a) and the magnetic hysteresis loops (b) of Fe-W composite coatings as a function of the applied current density in the plating bath. The apparent magnetization ranges from 184 to 200 emu/g, whereas the coercive field, H_c, varies between 10 and 15 kA/m. The composition of Fe-W composite coatings depends on W nanoparticles content in the plating bath. A higher content of W nanoparticles in the Fe matrix is obtained for a concentration of W nanoparticles in the plating bath of 40 g/l.

In Figure 9 are shown the temperature dependence magnetization curves (a) and the magnetic hysteresis loops (b) of Ni-W composite coatings as a function of the W content in plating bath.

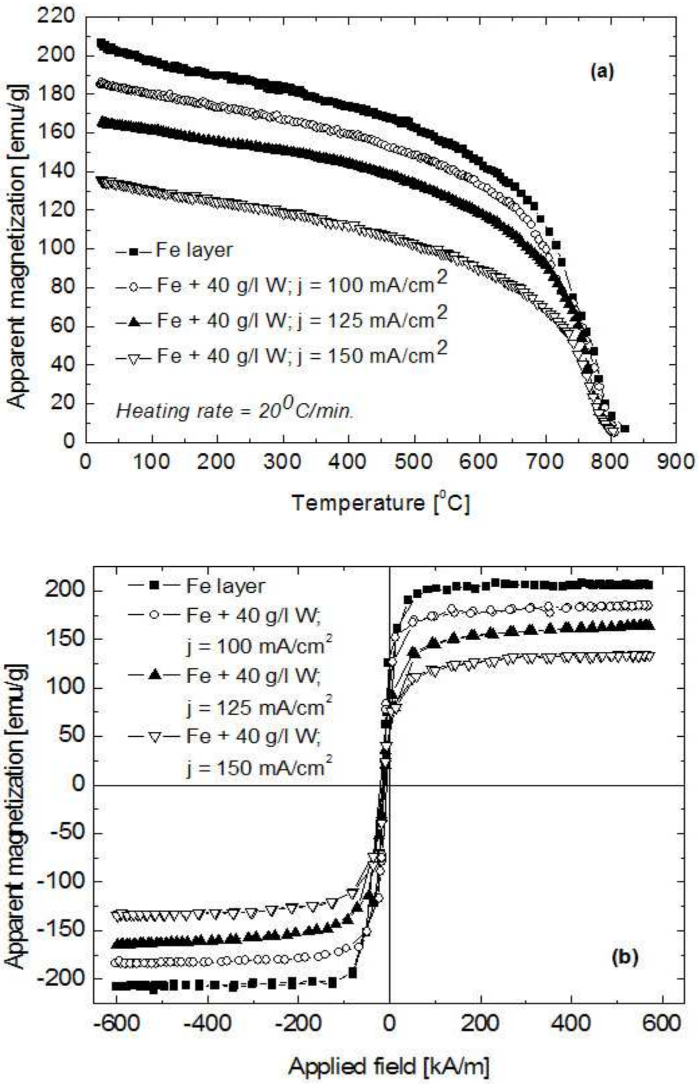

Fig. 8. Temperature dependence of the magnetization (a) and hysteresis loops (b) of Fe-W composite coatings, as a function of the current density in plating bath.

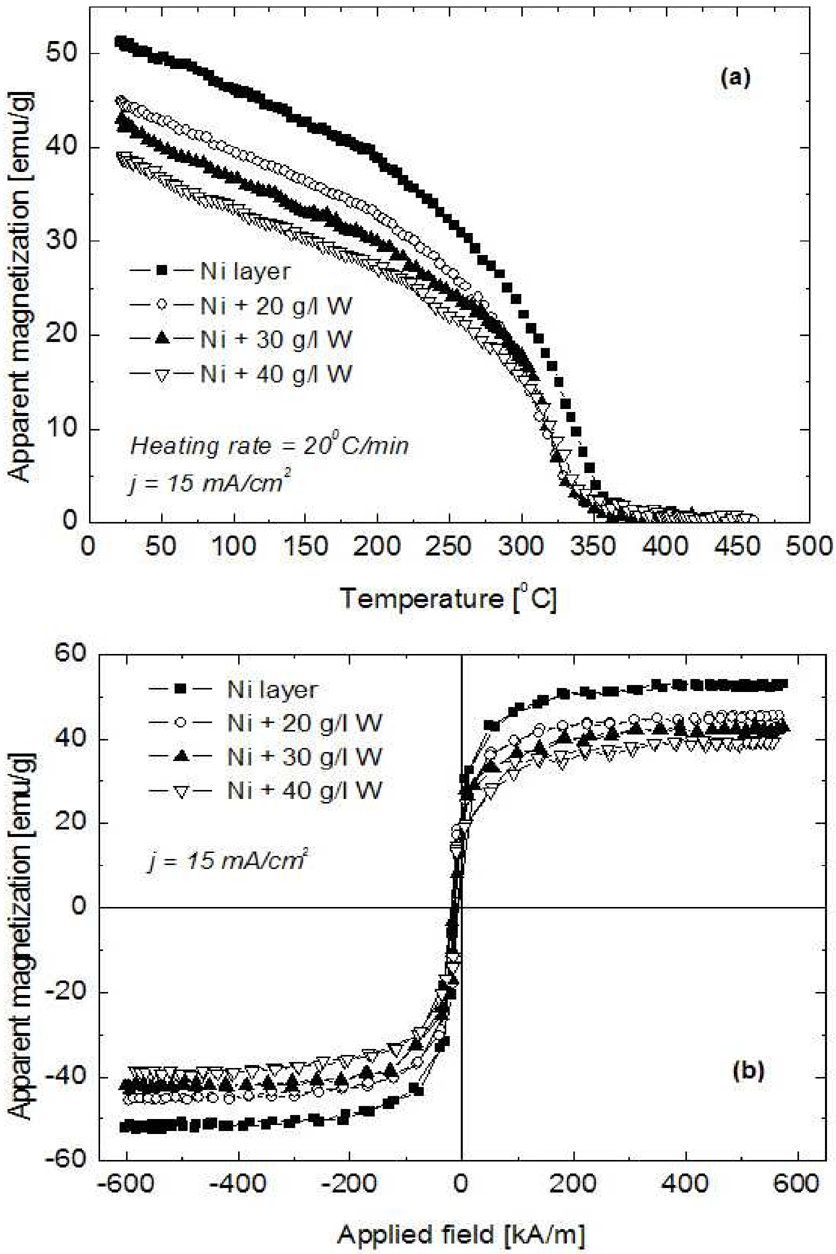

Fig. 9. Temperature dependence of the magnetization (a) and hysteresis loops (b) as a function of W content in the plating bath, for Ni-W composite coatings.

The thermomagnetic curves and magnetic hysteresis loops for Ni-W vomposite coatings, as a function of the current density applied in the plating bath are presented in Fig. 10. Ni-W composite coatings electrodeposited onto Au/Cr thin films exhibit apparent saturation magnetizations of 38–43 emu/g and coercivities between 10 and 13 kA/m. The apparent saturation magnetization of Ni-W composite coatings depends on the content of W nanoparticles in the plating bath.

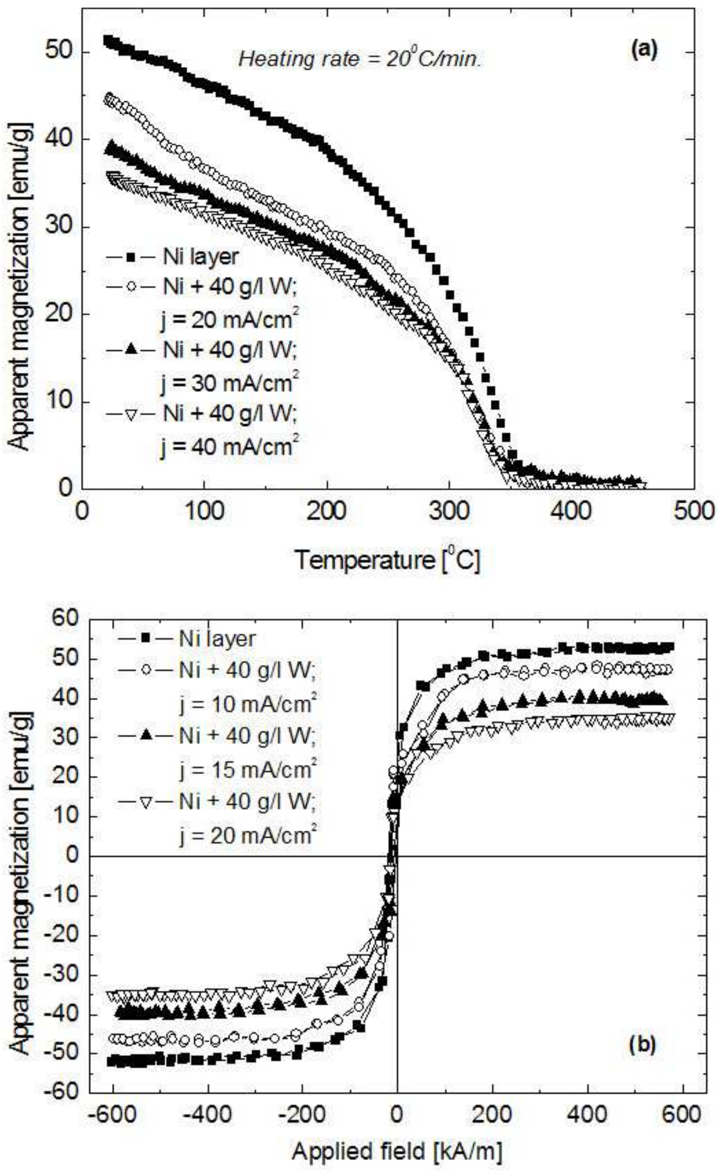

Fig. 10. Temperature dependence of the magnetization (a) and hysteresis loops (b) as a function of the applied current density in the plating bath, for Ni-W composite coatings.

4. Conclusions

Fe-W and Ni-W composite coatings can be electrodeposited from aqueous iron and nickel sulphate baths, respectively, containing W nanoparticles between 500 and 700 nm diameter. The obtained Fe-W and Ni-W composite coatings electrodeposited onto Au/Cr thin films present good magnetic properties. The principal parameters to assure a good uniformity of the embedded W nanoparticles in iron and nickel matrix during the co-deposition process are: the concentration of W nanoparticles in the plating bath, the current density, the deposition time and the mechanical stirring speed.

References

[1] LANDOLT D., J. Electrochem. Soc., **149(3)**, p. S9, 2002 .

[2] BALARAJU J. N., ANANDAN C., RAJAM K. S., Surface & Coatings Technol., **200**, p. 3675, 2006.

[3] POPCZYK M., SEREK A., BUDNIOK A., Nanotechnology, **14**, p. 341, 2003.

[4] SHAWKI S., HAMID Z. A., Aircraft Eng. Aerospace Technol., **69(5)**, p. 432, 1997.