

# Nanocomposites alumina/Pt produced by PVD and thermal oxidation methods used in catalytic applications

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## Abstract

The work was focused on the deposition of  $\text{Al}_2\text{O}_3 + \text{Pt}$  composite in the form of a nanofilm adherent to the substrate with good surface development and catalytic properties on the surface of 0H20J5 foil. With this end in view, a special carousel fixture for fixing the corrugated and flat foil and then coating with Al and Pt from two magnetron sources, in order to obtain a nanofilm of Al + Pt composite, was designed. A Pt/Al laminate layer was also produced by depositing Pt first and then Al, and the other way round. The layer systems obtained this way were subject to oxidation for 48 h at 860 °C and under  $\text{Ar} + \text{O}_2$ . The surface examined has shown that the process of oxidation produces the compact sublayer of columnar crystals (whiskers) well adherent to the substrate from which the needle-like whiskers are growing. Structures of whiskers form a laminar system with a catalytic element having a very-well developed surface. Structure analyses revealed that the films obtained comprised mostly  $\alpha$  and  $\theta$   $\text{Al}_2\text{O}_3$  phases and missed a Pt crystalline phase. The film morphology on the basis of oxide-film sections examined by TEM showed that the  $\alpha$   $\text{Al}_2\text{O}_3$  structure was generated mostly deep in oxide films and was integrated with the foil. A microanalysis showed that it was uniformly distributed in its entirety in the Pt-composite based layers; however, in the case of laminate the surface of whiskers was coated. It was found from the comparison of the laminar system activity that the composite layers revealed much better catalytic properties at lower temperature than the laminate layers.

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## 1. Introduction

Investigations on the oxidation of FeCrAl creep-resisting foils under  $\text{SO}_2 + \text{O}_2$  demonstrated that it is possible to obtain oxide films with very extensively developed surfaces and specified catalytic properties without the necessity of depositing the additional oxide [1,2]. Films with a similar character of surface development and similar catalytic properties were also produced by oxidising the foils twice longer but under more environmentally friendly  $\text{Ar} + \text{O}_2$  [3,4] conditions. An improvement in activity of the films deposited by thermal oxidation, especially due to the concentration of the basic sites, was obtained by modifying the surface with alumina nanofilms and enriching with Pt nanofilms additionally deposited by PVD methods [5]. It was found that the  $\theta$  phase content is decisive in increasing

the activity in  $\text{Al}_2\text{O}_3$  nanofilms, whereas the  $\alpha$  phase decreases this activity.

The work was focused on coating the surface of 0H20J5 foils with  $\text{Al}_2\text{O}_3 + \text{Pt}$  (composite) and  $\text{Al}_2\text{O}_3/\text{Pt}$  (laminate) nanofilms, with good adhesion to the substrate, showing activity and surface development that guarantee very good catalytic properties. The nanofilms were produced using metallic aluminium and platinum sputtered simultaneously from two DC magnetron sources on the surface of raw foil, or by depositing one film on the other in a bimetallic system. The film systems deposited in this way were subjected to the process of high-temperature oxidation.

## 2. Experimental procedures

### 2.1. Film deposition and thickness measurement

The substrate material designed for coating the films was foil in the form of thin flat and pleated sheets of 0H20J5

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Table 1  
Parameters of coating and specimen specification

|                       | Operating conditions for DC magnetrons with targets: |                      | Specimen specification                  |         |
|-----------------------|--|----------------------|---|---------|
|                       | Pt   | Al                   | Film systems                            | Symbol  |
| Base pressure         | 5.0E-5 mb  | 5.0E-5 mb            | Al on raw foil, oxidised                | Al      |
| Work pressure         | 3.0E-3 mb  | 3.0E-3 mb            | Pt on raw foil, oxidised                | Pt      |
| Flow rate of argon    | 2 cm <sup>3</sup> /s                                 | 2 cm <sup>3</sup> /s | Al/Pt laminate on raw foil, oxidised    | Al/Pt   |
| Power source          | 0.9 kW   | 0.6–0.9 kW           | Al + Pt composite on raw foil, oxidised | Al + Pt |
| Area of target        | 28 cm <sup>2</sup>                                   | 70 cm <sup>2</sup>   |   |         |
| Substrate temperature | 27 °C  | 27 °C                |   |         |

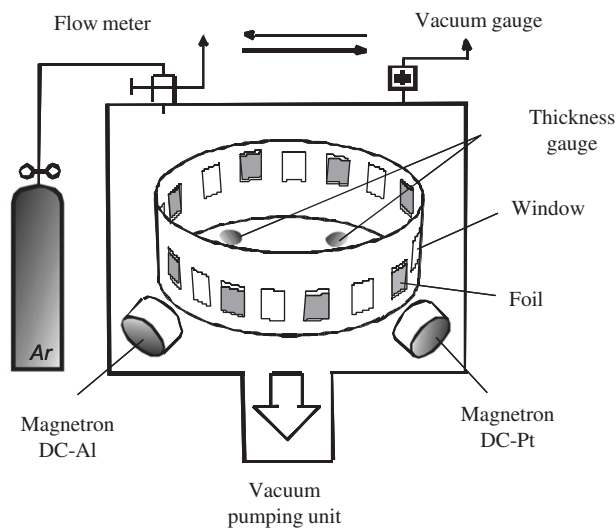


Fig. 1. Scheme of a vacuum chamber designed for deposition of Al and Pt nanolayers.

steel of the composition by weight: Cr 21.7%, Al 5.65%, Fe 72.45%, the rest of components 0.2%. Packets 12 mm in diameter and 70 mm in length designed for testing were composed of eight sheets 35 × 35 mm in dimension including four pleated sheets. The sheets were coated on both sides with Al and Pt composite nanofilms or bimetallic nanolaminate, where each of the components was sputtered by DC magnetrons. Working conditions for the processes are presented in Table 1. Magnetrons were arranged on a perimeter of a radius  $r_M = 200$  mm within the distance of  $l_M = 450$  mm. The sheets were mounted on the cylindrical holder  $r_C = 197$  mm in radius and  $l_C = 200$  mm in height, which had holes at half height with the shape and the area matched with the sheets. The sheets were between the holes from the outside, from the inside of the cylinder in front of magnetrons, and a quartz sensor for measuring the thickness was situated at the height of the holes (Fig. 1).

The sensor recorded an increase in the thickness of the film only if the hole appeared between the magnetron and the sensor. At the steady-state rotational speed of the

cylinder and the equal number of holes and sheets, the total thickness monitored by the sensor was matched to the thickness of the film deposited on each sheet. Thickness of the films was verified by the X-ray reflectivity method using a Philips' X'Pert instrument. The film systems obtained and their markings are compiled in Table 1.

The sheets coated with the films were oxidised for 48 h at the temperature of 860 °C under O<sub>2</sub> and Ar at a volume fraction 2:1 and a total flow  $V_f = 2,1$  ml s<sup>-1</sup>.

## 2.2. Examinations of structure

Changes in the surface of foil brought about by the process of oxidation were examined with a scanning electron microscope JSM 5500LV, applying the accelerating voltage 20 kV. The microstructure of films was examined with a transmission microscope Philips CM20 TWIN (200 kV) equipped with an EDAX Phoenix System that enabled it to detect and distribute the elements forming the film. With this end in view, thin foils were prepared by ion milling with Gatan Duomil 600 and then etched using the focused ion beam (FIB). The structure of composite films and metal-ceramic laminates was tested on a Siemens D5005 diffractometer, using the grazing incident X-ray diffraction method (GID) and CuK $\alpha$  radiation. The TOPAS (Bruker—AXS) programme was applied to a phase analysis.

The structure was also subjected to test on the nano-hardness of oxide and composite films using a Hysitron Incorporated Nanoindenter. The test was carried out with a Berkovich's indenter with loads applied continuously between 0 and 5000  $\mu$ N.

## 2.3. Catalytic test

To determine the catalytic activity of the deposited film systems and the oxide-latinum composites, the acidic and basic sites and their share in processes of conversion and dehydration of alcohol, which was 2-methylol-3-butyn-2-yn, were identified. The test of catalytic activity was carried out by the pulse method in the 200–350 °C temperature bracket [5,6].

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