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Reactive Chemical Vapour Deposition of titanium carbide from H_2 - $TiCl_4$ gas mixture on pyrocarbon: A comprehensive study

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Abstract

In Reactive Chemical Vapour Deposition (RCVD), the absence of one element of the deposited carbide in the initial gas phase involves the consumption/conversion of the solid substrate. In this way, the growth of a continuous carbide layer on the substrate requires solid-phase diffusion of the reagent.

In this work, a parametric study of the RCVD of titanium carbide from pyrocarbon (PyC) and an H_2 - $TiCl_4$ mixture has been carried out. Conversion ratio, PyC consumption and carbide layer growth kinetics have been determined at 1000°C. The influence of the $H_2/TiCl_4$ dilution ratio has been also investigated. The apparent inter-diffusion coefficient of the carbon through the TiC deposited layer and the direct apparent reaction rate were determined from a comparison between simulations based on a Deal-Grove-type model and the experimental results. The study has been completed with FTIR spectrometry analyses of the gases.

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Keywords: CVD ; R-CVD ; TiC coating ; pyrocarbon ; solid state diffusion ; FTIR ; simulation

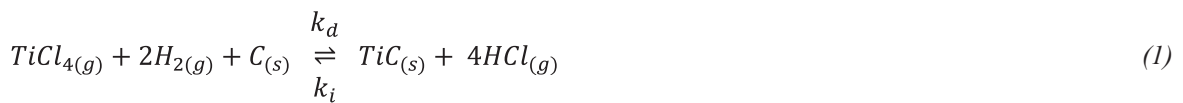
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Nomenclature	
k_d	Direct rate constant ($\text{mol.m}^{-2}.\text{s}^{-1}$)
k_i	Inverse rate constant ($\text{mol.m}^{-2}.\text{s}^{-1}$)
r	Dilution ratio of TiCl_4 in H_2
x	Carbon content in the TiC
D_C	Solid-state apparent inter-diffusion coefficient of carbon in TiC ($\text{m}^2.\text{s}^{-1}$)
D_{TiCl_4}	Gaseous diffusion coefficient of TiCl_4 ($\text{m}^2.\text{s}^{-1}$)
$p_{\text{TiCl}_4,s}$	TiCl_4 partial pressure on the deposition surface (Pa)
$p_{\text{TiCl}_4,\infty}$	TiCl_4 partial pressure in the RCVD reactor (Pa)
$p_{\text{H}_2,\infty}$	H_2 partial pressure in the RCVD reactor (Pa)
v_r	Reaction (I) rate ($\text{mol.m}^{-2}.\text{s}^{-1}$)
j_C	Carbon flux from the PyC layer across the deposited TiC layer ($\text{mol.m}^{-2}.\text{s}^{-1}$)
j_{TiCl_4}	TiCl_4 flux from the bulk of the reactor across the limit diffusion layer ($\text{mol.m}^{-2}.\text{s}^{-1}$)
$V_{m,C}$	Carbon molar volume ($\text{m}^3.\text{mol}^{-1}$)
$V_{m,\text{TiC}}$	TiC molar volume ($\text{m}^3.\text{mol}^{-1}$)
c_0	Reference concentration of the TiC ($1/V_{m,\text{TiC}}$) (mol.m^{-3})
a_i	Interfacial activity of carbon (interface between TiC and PyC)
a_s	Carbon activity in the gas phase near the reaction surface
R	Perfect gas constant ($\text{J.mol}^{-1}.\text{K}^{-1}$)
T	Temperature (K)
θ	Temperature ($^{\circ}\text{C}$)
δ	Limit diffusion layer (m)
P_0	Reference pressure (Pa)
$R_{v,th}$	Theoretical conversion ratio
$R_{v,ap}$	Apparent conversion ratio
e_{TiC}	TiC thickness (meters for the modeling part and nanometers for the experimental part)
$e_{C, cons}$	PyC consumed thickness (nm)
$e_{C, ini}$	Initial PyC layer thickness (nm)
$e_{C, res}$	Residual PyC layer thickness (nm)

1. Introduction

Titanium carbide (TiC) is a frequently studied material for nuclear (Groot et al., 1991) and aerospace engineering applications (Boving and Hintermann, 1990). This material is extremely hard, has good corrosion resistance and refractoriness and is highly electrically conducting. These characteristics made him a good material for being studied.

The Reactive Chemical Vapour Deposition (RCVD) method is used to form carbide coatings and allows thin, homogeneous and adherent layers to be obtained. For instance, carbides (HfC, TiC ...) and boro-carbides (B_4C ...) from metals of groups 4 and 5 have been deposited for barrier applications in refractory ceramics (Piquero et al., 1995, Baklanova et al., 2006). These authors mentioned that RCVD coating are thin, homogeneous and adherent. The purpose of this work was to study the RCVD of titanium carbide from pyrocarbon according to the overall reaction (I).



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