MULTIPROTECT

SIXTH FRAMEWORK PROGRAMME

SiO_2 based hybrid inorganic-organic films doped with oxide nanoparticles for corrosion protection

Powders preparation

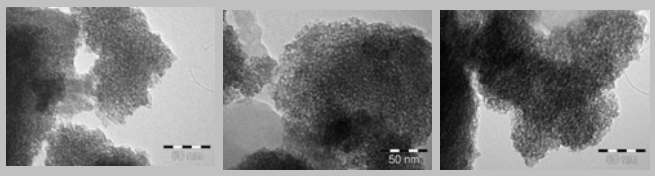
Monocomponent and binary nanosized powders

Sample	Composition	Reagents	Particle Size
TiO_2	100	$Ti(OC_2H_5)_4$	10 nm
ZrO_2	100	$Zr(OC_3H_7)_4$	20 – 50 nm
CeO_2	**100**	**$Ce(NO_3)_3.6H_2O$**	**10 – 50 nm**
CeO_2	100	$Ce(CH_3CO_2)_3.xH_2O$	3 – 5 nm
$ZrO_2 – CeO_2$	80 : 20	$Zr(OC_3H_7)_4$, $Ce(NO_3)_3.6H_2O$	~ 10 nm
$ZrO_2 – CeO_2$	**80 : 20**	**$ZrO(NO_3)_4$, $Ce(NO_3)_3.6H_2O$**	**1 – 2 nm**
$TiO_2 – CeO_2$	**80 : 20**	**$Ti(O\text{-}iC_3H_7)_4$, $Ce(NO_3)_3.6H_2O$, NH_4OH**	**2 – 3 nm**
$TiO_2 – CeO_2$	80 : 20	$Ti(O\text{-}iC_3H_7)_4$, $Ce(NO_3)_3.6H_2O$, urea	~10 nm
$TiO_2 – CeO_2$	**50 : 50**	**$Ti(O\text{-}iC_3H_7)_4$, $Ce(NO_3)_3.6H_2O$, NH_4OH**	**2 – 4 nm**
$TiO_2 – CeO_2$	80 : 20	$Ti(O\text{-}iC_3H_7)_4$, $(CH_3CO_2)_3Ce.xH_2O$	5 – 10 nm
$TiO_2 – CeO_2$	50 : 50	$Ti(O\text{-}iC_3H_7)_4$, $(CH_3CO_2)_3Ce.xH_2O$	5 nm
V_2O_5-CeO_2	**50 : 50**	**V_2O_5, $Ce(NH_4)_2(NO_3)_6$**	**10-20 nm**

Drop-test results for the most significant powders

Sample	24h	48h	72h
CeO_2	1	3	5
ZrO_2-CeO_2	5	6	6, surface pasivation
TiO_2-CeO_2 (80:20)	3	3	0, surface pasivation
TiO_2-CeO_2 (50:50)	3	4	6
V_2O_5-CeO_2 (50:50) dried	1	2	2
V_2O_5-CeO_2 (50:50) thermally treated	5	5	7

Reproducibility test of powder preparation

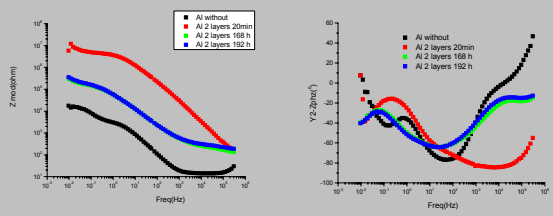


TEM micrographs for TiO_2-CeO_2 powders: 80:20 thermally treated at 400 °C

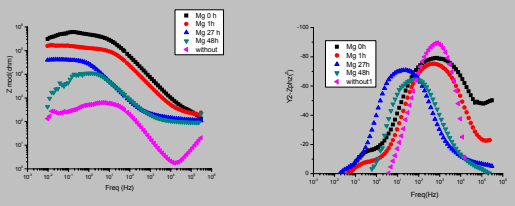
Coatings preparation

Experimental conditions used for coatings preparation by dip method

- Matrix composition: 65%TEOS+35%TSPM
- Coatings composition: matrix with addition of 5-10% non-functionalized binary [80%TiO_2(ZrO_2)-20%CeO_2] previously prepared powders
- Deposition of the coatings was realized by dip coating with a withdrawal rate of 5 /min. on Al2024 and Mg substrates. One or two layers were deposited
- Thermal treatment of the obtained films was realized at 120 for 2 hours



EIS Bode for Al2024 alloys coated with silica based matrix and TiO_2-CeO_2 powder immersed in NaCl 0.05 M, as compared with the uncoated substrate (determined at Instituto de Cerámica y Vidrio (ICV), Madrid, Spain)



EIS Bode for Mg alloys coated with silica based matrix and TiO_2-CeO_2 powder immersed In NaCl 0.05 M, as compared with the uncovered substrate (determined at ICV)

Third functions results (determined at CASTI)

Sample	Contact angle	Scrach, g	Adhesion
Coating deposited in Al2024	73,3 ± 2,3	850 ± 50	5B (0%)
Coatings deposited on Mg ZK10	72 ± 3	380 ± 50	5B (0%)
Coatings deposited on Mg AZ31	73 ± 6	260 ± 50	5B (0%)

Results on demonstrators

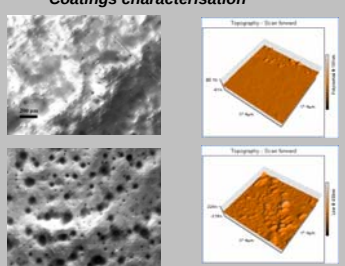
Conditions used at robotic spraying by Fraunhofer Gesellschaft, IPA Stuttgart, Germany

V	Thickness µ
150	14,9
300	6,9
400	4,8
600	3,1

Suspension used and coatings obtained on demonstrator

Sol-gel coating	Film thickness	Curring conditions	Viscosity	Surface tension
SiO_2-based hybrid matrix doped with binary TiO_2-CeO_2 powder	7 µm (5-10 µm)	120°C, 2h	3.1 (mPass)	23.6 (mN/m)

Coatings characterisation



SEM and AFM images of the coatings deposited by robotic spraying at IPA (V300-up: V400-down)

Corrosion tests (measured at Deutschland GmbH, Corporate Research Center Germany (EADS), Munich, Germany

Q-Panel condensation Test

➢No delamination or blistering after 500 hours test duration

AA2024 AA7475

Salt spray test bare (168 h)

> 100 pits after 168h

AA2024 – 168h AA7475 – 168h

Salt spray test painted (500 h)

AA2024 AA7475

Filiform corrosion test (500 hours)

AA2024 AA7475

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