# The 30th International Conference on Surface Modification Technologies

## PROGRAM

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<td>Opening Ceremony</td>
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<td>9:00-9:40</td>
<td>PLENARY LECTURE: Prof. S. A. Meguid</td>
<td>PLENARY LECTURE: Prof. J. T. M. de Hosson</td>
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<td>PLENARY LECTURE: Prof. K. Lu</td>
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<td>Laser based processes 3</td>
<td>Surface wettability</td>
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<td>18:00-19:00 WELCOME PARTY (CIVICO MUSEO ARCHEOLOGICO DI MILANO)</td>
<td>19:00-22:00 GALA DINNER (CASTELLO SFORZESCO DI MILANO)</td>
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**BL.27.04:** Plenary lectures & Sessions  
**BL.27.05:** Sessions  
**BL.27.06:** Sessions  
**BL.27.11:** Sessions
9:40-11:00

**Laser based processes 1 (BL.27.04)**
Chairman: P.Carbone
319 (keynote): J.L.Ocaña; J.A.Porro; M.Díaz; L.Ruiz-de-Lara; D.Peral; J.A.Santiago; I.Angulo; “Laser shock processing: an emerging technique for the mechanical and surface properties enhancement of metallic materials”
097: F.Caiazzo; V.Alfieri; G.Corrado; P.Argenio; V.Sergi; “Laser surface modification by means of scanning optics on stainless steel molds for investment casting”
168: C.Rotty; M.-L.Doche; A.Mandroyan; J.-Y.Hihn; G.Montavon; “Electropolishing of thermal spray coating and laser additive manufacturing of 316L stainless steel in strong acidic media”
132: U.A.Kumar; A.Priyadarshi; S.Arvindan; “Localised boronation of armour steel using tungsten arc”

**Surface modification 1 (BL.27.05)**
Chairman: M.H.Staia
179 (keynote): J.Song; H.Yuan; C.Koch; E.Silberngel; V.Schinow; “Tribological properties and fretting performance of gold, tin and silver coatings”
146: S.Hanke; J.Sena; R.S.Coelho; J.F.dos Santos; “Microstructural features of dynamic recrystallization in Alloy 625 friction surfacing coatings”
175: V.S.Sergeyev; I.V.Blinkov; A.O.Volkhonski; D.S.Belov; “The effect of Ni on structure and properties of adaptive wear-resistant arc-PVD Ti-Al-Mo-N coatings”
307: S.M.Yunus; M.J.Ghazali; A.A.Pauzi; W.F.H.W.Zamri; “Comparative wear characteristics of chromium-carbide and chromium-cobalt coatings for combustor liners”

**Shot peening 1 (BL.27.06)**
Chairman: A.Zammit
349 (keynote): L.Wagner; M.Wollmann; “Shot peening induced improvements of the fatigue performance of light alloys based on titanium, aluminum and magnesium”
074: M.Benedetti; V.Fontanari; M.Aliakbari; J.C.Hanan; M.Bandini; “An area method to incorporate roughness and residual stress effects on plain and notched fatigue behavior of shot peened aluminum alloys”
060: P.Q.Trung; D.L.Butter; N.W.Khun; “Three-dimensional dynamic finite element modelling of shot peening process: new approach to estimate level of the coverage parameter”
101: A.H.Mahmoudi; F.Salahii; A.Ghasemi; “A comparison between residual stresses induced by water jet peening and shot peening for surface modification”

9:40-11:00

**Laser based processes 2 (BL.27.04)**
Chairman: B.Previtali
295 G.Daurelio; A.Angelastron; S.L.Campanelli; L.A.C.De Filipis; A.Pugliese; A.D.Ludovico; “Fiber laser remelting and surface hardening of a Nickel based superalloy”
336: V.Furlan; A.G.Demir; C.Paternoster; R.Tolouei; D.Mantovani; B.Previtali; “Surface oxidation by laser soft-melting treatment to change degradation behaviour of a magnesia alloy”
208: R.Kromer; S.Costil; D.Moskali; S.Houdková; “Laser treatments (ns, ps) on aluminium substrate to create hydrophobic surfaces for molding process”
149: A.Kanjer; Z.Wu; P.Berger; V.Optasanu; S.Dejardin; M.C.Marco de Lucas; M.François; T.Montesin; L.Lavisse; “Retarding the high temperature oxidation of Titanium by surface Laser treatment”

**Surface modification 2 (BL.27.05)**
Chairman: M.Benedetti
266: M.Broivy; E.Privas; S.Bistac; “Surface and friction properties of anti-sticky fluorinated functional coatings”
279: M.H.Staia; A.Trocelis; A.Zairi; M.Suarez; E.S.Puchi-Cabrera; A.Lost; A.Montagné; “Assessment of the mechanical, tribological and corrosion performance of a ZrN PVD coating”
148: M.Benedetti; V.Fontanari; C.Girardi; L.Giardano; “Alternative solutions to the electroplating corrosion inhibition of steels: a comparative study of different surface treatments of steel”
252: W.Tillmann; L.Hagen; D.Kokalj; M.Paulus; M.Tolan; “Influence of high-temperature oxide formation on the tribological behavior of Fe-V arc sprayed coatings”

**Shot peening 2 (BL.27.06)**
Chairman: L.Wagner
147 (keynote): A.Kanjer; Z.Wu; P.Berger; V.Optasanu; S.Dejardin; M.C.Marco de Lucas; M.François; T.Montesin; L.Lavisse; “Retarding the high temperature oxidation of Titanium by shot peening surface treatment”
163: K.McClurg; F.Chateau; L.Wagner; “Expanded Fatigue Qualification for Ultrasonic Shot Peening”
318: M.Gholami; J.Altenberger; H.A.Kuhn; M.Wollmann; L.Wagner; “Effect of shot peening on fatigue performance of ultralight-grained CuNi35Si1Mg alloy”
155: A.Bonadei; C.Calcagno; G.Delugan; A.Zerah; “Influence of the shot peening parameters on microstructure in Renè80 Nickel-based superalloys”

11:00-12:00

**COFFEE BREAK**
Severe plastic deformation (BL.27.04)
Chairman: K. Lu
020 (keynote): S. Bagherifard; “Severe shot peening: a path towards multifunctional metallic biomaterials”
161: M. Cao; Q. Zhang; Y. Zhang; “Surface modification of tin copper alloy components by integrate rotary swaging and isothermal heat treatment”
340: Y. Zhang; B. Han; Q. Zhang; M. Cao; X. Yang; H. Yang; “FEA and experiment study on joining by rotary swaging method”

Thermal spraying (BL.27.05)
Chairman: B. Rivolta
095 (keynote): G. Mauri; L. Du; R. Vášen; “Atmospheric plasma spraying of single phase lanthanum zirconate thermal barrier coatings with optimized porosity”
264: J. Kotlan; R. Musalek; J. Medricky; T. Tesar; F. Lukac; T. Chraska; “Suspension Plasma Spraying of YAG Using WSP®H - High Enthalpic Plasma Torch”
284: J. Kotlan; R. Musalek; J. Medricky; T. Tesar; F. Lukac; T. Chraska; “Low Cycle Fatigue of Seam Welds – Numerical Simulation under Consideration of Material Inhomogeneities”

Shot peening 3 (BL.27.06)
Chairman: A. H. Mahmoudi
156 (keynote): A. Zammit; M. Bonnici; M. Mhaede; R. Wan; L. Wagner; “The effect of Shot Peening on Austempered Ductile Iron Gears”
182: K. Nambu; K. Monda; K. Inagaki; Y. Maeyama; S. Kikuchi; “Effect of hardness ratio on the behavior of plastic deformation in various metallic materials treated with fine peening”
177: D. Grell; L. Böhme; S. Dietrich; J. Gibmeier; F. Silze; U. Kühn; V. Schulze; E. Kerscher; “Influence of shot peening on the mechanical properties of bulk metallic glass Vitreloy 105”

Laser based processes 3 (BL.27.04)
Chairman: J. L. Ocaña
084 (keynote): S. Valette; N. Pionnier; S. Hammouti; J. Dufils; V. Belaud; E. Contraires; R. Berger; S. Benayoun; “Surface functionalization through femtosecond laser multiscale texturing: examples through anti-fog, anti-ice and anti-wear functions”
309: R. Jagdheesh; M. Diaz; J. L. Ocaña; “Robust transformation of wetting properties of metallic surface by ns laser”
322: D. Verdi; C. I. Múñez; M. A. Garrido; P. Pozza; “Improving the performance of laser cladded inconel 625 coatings by the addition of Cr₃C₂ particles”
312: J. M. López López; J. Coupland; S. Marimuthu; “Droplet assisted laser cleaning of contaminated surfaces”

Surface wettablity (BL.27.05)
Chairman: M. P. Pedederi; M. V. Diamanti
302 (keynote): M. V. Diamanti; C.-Y. Lai; M. Cozzolino; M. Pedederi; S. A. Hassan; M. Chiesa; “Time-dependent surface wettablity of calcite: a baseline for oil reservoirs investigations”
343: L. Li; S. Fan; C. Zhuang; J. Feng; C. Ban; X. Liu; “Controllable wettablity of boron nitride nanotube films with bionic effect”
202: C. Garlisi; G. Scandura; Y. H. Chang; C. Y. Lai; T. Olukun; M. Chiesa; G. Palmisano; “Investigation on the wettablity of TiO₂ evaporated thin films by atomic force microscopic and contact angle”
120: L. Yin; H. Zhang; R. Weng; R. Zhang; X. Liu; “Design and characterization of MEMS rotational gyroscope with superoleophobic surfaces”

Shot peening 4 (BL.27.06)
Chairman: S. Bagherifard
191: Y. Kameyama; Y. Fujioka; H. Okada; K. Endo; H. Sato; R. Shimpoo; “Effect of hybridized shot particles fabricated by mechanical milling on the material transfer during peening process”
234: J. P. Fuhr; M. Basha; M. Wollmann; L. Wagner; “Coverage and peening angle effects in shot peening on HCF performance of Ti-6Al-4V”
285: C. Lagó; M. Guagliano; O. Bokóvka; L. Trško; O. Řídký; F. Nový; D. Závodská; “Improvement of fatigue endurance of welded S355 J2 structural steel by severe shot peening”
327: F. Yin; M. Rakita; Q. Han; “Overview of ultrasonic shot peening”

Laser Coatings 3 (BL.27.11)
Chairman: N. Cinca
194: H. Jung; J. S. Park; H. Kim; “Growth behaviors and electrical properties of high-k films by using pulsed PE-CVD and PE-ALD”
331: L. Aissani; M. Fellah; C. Nhouveu; H. Djebaili; “The effect of annealing treatment and nitrogen pressure in the formation of chromium nitrides, carbides and carbonitrides coatings”
216: S. Baragetti; R. Gerosa; F. Villa; “PVD DLC coatings for corrosion protection on a 7075-T6 substrate at long and short fatigue lives”

Surface coatings for corrosion protection on a 7075-T6 substrate at long and short fatigue lives
### Thursday, June 30th

| 8:00-9:00 | Registration |
| 9:00-9:40 |PLENARY LECTURE (BL.27.04): Prof. J.T.M. de Hosson |

#### 9:40-11:00

| 9:40 | Residual stresses 1 (BL.27.04) Chair: M.Sebastiani 154: I. Guenolé; E. Bitzek; “Atomic simulations of focused ion beam machining and resulting irradiation damage induced eigenstrains in metals and ceramics” 145: D. Courty; A.S. Sologubenko; S.S.A. Gerst; R. Spolenak; “Ga-implantation as a factor limiting the evaluation of the material stress state by FIB” 213: C. Schmid; E. Bruder; J. Dluhoš; R. Váňa; L. Benker; M. Göken; K. Durst; “Stress relaxation during FIB milling assessed by digital image correlation, in situ micro-Raman spectroscopy and electron backscatter diffraction” 088: S. Ben Afia; D. Retract; B. Panicaud; J.L. Grosseau-Poussard; “Comparison of growth stresses developed in a thermally grown oxide on a nanocrystallised stainless steel, evaluated by in-situ Raman spectroscopy and numerical simulations” |
| 10:00 | Ti surface treatments (BL.27.05) Chairman: M.V. Diamanti 281: E. Marin; A. Lanzutti; A. Raffaelli; M. Magnan; M. Regis; R. Offoach; F. Sbai; L. Fedrizzi; “Induction nitriding of grade 5 titanium alloy: process optimization” 119: L. Skowronski; “Fabrication and characterization of TiO$_2$/Ti-based decorative coatings produced at industrial scale using the magnetron sputtering technique” 121: D.I. Adebiyi; O. Fatobab; S. Pityana; P. Popoola; “Parameters optimization, microstructure and micro-hardness of silicon carbide laser deposited on titanium alloy” 102: M. Liberini; A. El Hassanin; G. De Falco; M. Commodo; A. D’Anna; A. Squillace; L. Carrino; “Properties and characteristics of nano-TiO$_2$ coatings produced by a dynamic system through the spray flame synthesis” |
| 10:20 | Cold Spray 1 (BL.27.06) Chairman: H. Koivuluoto 066 (keynote): I. Villafuerte; “Modern Cold Spray Future Trends” 150: M. Robotti; S. Dosta; C. Fernández-Rodríguez; M.J. Hernández-Rodríguez; I.G. Cano; E. Pulido Milán; J.M. Guillemay; “Photocatalytic abatement of NOx by C-TiO$_2$/Polymer composite coatings obtained by Low Pressure Cold Gas Spraying” 091: J. Cizek; L. Cenev; M. Matejkova; J. Kouril; J. Cuperas; I. Dluhy; “Potential of new-generation electron beam technology in interface modification of cold and haf sprayed Mcraly bond coats” 207: R. Kromer; Y. Danlos; S. Costil; D. Moskal; S. Houdková; “Coating adhesion enhancement by laser surface texturing - deposition characteristics of metallic particles on different classes of substrates in cold spraying process” |

#### 11:00-11:20 

**COFFEE BREAK**

#### 11:20-13:00

| 11:20 | Residual stresses 2 (BL.27.04) Chair: M.Sebastiani 159: M. Renzelli; Z. Mughal; M. Sebastiani; E. Bemporad; “Optimization of coating scratch resistance trough computationally designed stress profiles” 165: M.Z. Mughal; P. Hofmann; K. Fadenberger; M. Sebastiani; “Design and Characterization of a novel Chromium Nitride (CrN) coating for application in diesel injection systems” 085: E. Auerswald; D. Vogel; B. Michel; S. Rzepek; “Local measurement of residual stresses in thin layers” 198: A.M. Korsunsky; E. Salvati; H. Zhang; T. Sui; “Mechanical microscopy of surface modification induced by electro-discharge machining” 334: I. Hermann; B. Ohler; “Quantitative nano-mechanical characterization of composites and thin films” |
| 11:40 | Coatings funct/conv (BL.27.05) Chairman: F. Libonetto 100: A. Ponsoni; S. Ottoboni; M. Guilizzoni; M. Zani; P. Gronchi; “Properties of steel surfaces coated with organic molecules” 245: A. Astarita; S. Genna; C. Leone; M. Liberini; A. Squillace; “On the influence of different superficial laser texturing on the deposition of powders through cold spray process” 243: G. Baiocco; M. Simoncini; V. Tagliaferri; N. Ucciardello; “Combined electrodeposition of copper and graphene nano-platelets on aluminium’s alloys substrate for corrosion protection” 173: D. De Felicis; M. Renzelli; E. Bemporad; “Mechanical and color stability of black multilayer Ti-C-N thin film prepared by reactive - magnetron sputtering” 305: E. Karacam; F. Muhaffel; M. H. Zamani; M. Divandari; H. Cimenoglu; “Formation of a protective ceramic coating on friction stir welded magnesium and aluminum by micro arc oxidation” |
| 11:40 | Surface processes (BL.27.11) Chairman: M. Bandini 153: C. Langlade; A. Roman; D. Schlegel; E. Gete; M. Folea; “Fiction stir process of B194 cooper-beryllium alloy” 339: D. Závodská; E. Tillovo; M. Guagliano; L. Kuchariková; M. Chalupová; J. Lago; “Fatigue resistance of self-hardening AlZn10Si8Mg alloy” 218: T. Czerwiec; A. Andrieux; G.A. Bortolini; P.H. Bolzan; G. Marcos; “Evaporation of liquid sessile drops on polished and patterned metal surfaces for thermal management” 103: M.V. Loureiro; M.J. Lourenço; A. De Schrijver; J.C. Bordado; A.C. Marques; “Amino surface functionalized microcapsules as curing agents for polyurethane foams” 315: G. Boletti; A. Candel; L. Lusvarghi; A. Ravaux; A. Denoirjean; S. Valette; C. Chazelas; E. Meillot; L. Bianchi; “NiCrAlY + Al$_2$O$_3$ + h-BN composite coatings by "hybrid" plasma spraying” |
Corrosion (BL.27.04)
Chairman: A.Astarita

310 (keynote): S.L.Ruiz-de-Lara, R.Jardheesh, J.L. Ocaña; "Corrosion behavior of superhydrophobic aluminum surfaces"
286: D.Fontozzi; V.Matikainen; M.Uusitalo; H.Koivuluo; P.Vuristo; “High-temperature corrosion resistance of HVOF and HVAF sprayed Cr2C3-based coatings under KCl salt deposit”
200: V.Genova; F.Marra; L.Fedrizzi; A.Lanzutti; C.Bartuli; T.Valente; G.Pulci; “Droplet assisted laser cleaning of contaminated surfaces”
098: S.Rossi; M.Fedel; L.Da Col; F.Deflorian; S.Petrolli; “Coatings deposition to increase the corrosion behaviour of aluminium foam”
268: E.Marin; A.Lanzutti; L.Fedrizzi; “Improving metal corrosion resistance by atomic layer deposition”
099: P.Gronchi; R.Canziani; A.Brenna; S.Visigalli; C.Colominas; F.Montalà; V.Cot; A.Stradi; G.Ferrari; V.Bellelli; C.Diaz; G.Garcia Fuentes; “Evaluation of electrode surface treatments in sludge electro-erosion dewatering”

Applications (BL.27.05)
Chairman: S.Bahgerifard

193: M.Winnicki; A.Malachowska; M.Korzeniowski; M.Rutkowska-Gorzyczka; “The influence of interlayer thickness on mechanical properties of aluminium to steel resistance spot weld”
257: A.Carangelo; M.Curioni; T.Monetta; F.Bellucci; “Alternatives to chrome-based coating for aerospace aluminium alloys anodized by the tartaric-sulfuric acid process”
067: S.Pathak; N.K.Jain; I.A.Palian; “Study on surface imperfections of gears: sources, effects, and techniques for concerned improvements”
090: N.Babcsan; B.Gastón; G.Garrido; J.M. Vega; G.Vara; “Electropolishing as a pretreatment for anodizing and plasma electrolytic oxidation (PEO) of aluminium matrix composites and aluminium foams”
344: I.Cerny; I.Fürbacher; D.Mikulová; J.Sis; N.Ganev; K.Kolarik; “POTENTIAL INCREASE OF GEAR SERVICE LIFE USING LASER HARDENING TECHNOLOGY”
346: C.Bhowmik; A.Ray; S.Bhowmik; “Selection of energy efficient material: an entropy-topsis approach”

Cold Spray 3 (BL.27.06)
Chairman: P.Pozza

110: H.Koivuluo; M.Honkanen; D.Fantozzi; M.Vippola; P.Vuristo; “Microstructural Characteristics and Corrosion Properties of Cold-Sprayed Incnel 625 and 718 Coatings”
057: N.M.Chavan; L.Venkatesh; P.Suresh Babu; P.Pant; G.Sundararajan; “Cold sprayed copper and copper aluminum alloys: influence of stacking fault energy on the structure and properties”
267: S.Buhl; P.Breuninger; S.Antonyuk; “Optimization of a laval nozzle for energy efficient cold spraying of microparticles”
123: S.Borhan Dayani, R.Ghelichi; H.Jahed; “Corrosion-fatigue endurance of cold spray coated AZ31B magnesium alloys”
321: P.Sirvent; M.A.Garrido; C.J.Müñez; P.Pozza; A.Cazacu; R.Barnett; H.L.de Villiers Lovelock; “Effect of powder geometry on the mechanical performance of cold sprayed Ti-6Al-4V coatings”
350: J.Kondas; “Advanced high pressure cold spray coatings and selected applications”

Lunch

13:00-14:20

14:20-16:00

Poster session

059: W.M.Roschin, I.N.Petukhov, A.Savateeva, M.S.Vagin, A.V.Roshchina; “Technology application of non-crystalline carbon coating deposition by pulsed arc plasma”
061: P.Q.Trung; N.W.Khun; D.L.Butler; “Comparison of the effects of conventional shot peening and severe shot peening processes on the mechanical and tribological properties of shot peened AISI 4340”
064: M.Weiser; C.Schulze; A.Meyer; A.Potthoff; M.Schneider; “Characterization of the electrolytic deposition of particle reinforced metal electrolyses by electrochemical quartz crystal micro balance”
075: M.Parvizian; F.Rahimi-Ashkari; A.Goodarzi; “The effect of radio-frequency sputtering power on structural properties of Chromium thin films”
078: Y.-S.Yang; Y.-J.Lin; C.-Y.Chen; “Effect of Ti-N and Cr-N period number on the corrosion and wear properties of Ti-N/Cr-N Multilayer coatings”
080: D.Qin; T.Gao; H.Wang; X.Peng; Z.Zheng; “Effect of process parameters on energy consumption in radial-axial ring rolling process”
082: Z.Fan; K.Wang; X.Dong; R.Wang; W.Duan; X.Mei; W.Wang; S.Zhang; “Effect of ultrasonic vibration on microstructure and corrosion resistance of laser re-thermal melt deposited TBCs”
089: M.Scendo, K.Staszewska, B.Antoszewski; “Corrosion Protection of DLC Coating on Steel Surface”
113: M.J.Miraizal; V.Mussi; F.Libonati; M.Strano; L.Vergani; “Manufacturing Process of a New Aluminum Foam Material Inspired to Tribecular Bone”
117: R.Wang; X.Dong; Z.Fan; K.Wang; W.Duan; X.Mei; W.Wang; J.Cui; “Effect of laser remelting assisted with induction preheating on microstructure and thermal shock resistance of laser remelted TBCs”
118: J.Cao; J.Zhou; Y.Zhang; X.Liu; “Microwave-Assisted Synthesis of GCO Surface Supported MoS2 Catalyst for Hydrogen Evolution Reaction”
141: C.Leong; H.Kim; J.Park; M.Lee; Y.Jeon; “FEF Simulation and Experimental Validation of Residual Stress on Surface by Micro Forging Processes.”
166: C.Rotty; M.-L.Doche; A.Mandroyan; S.Monney; S.Lallemand; J.-Y.Hihn; “Roughness characterization procedures for electropolished brass”
172: L.Escalada; A.Gasco Owens; E.L.Dallison; G.Marcos; T.Czerwiec; S.P.Bruhl; S.N.Simison; “Corrosion and tribological performance of low-temperature carburized austenitic stainless steel. Role of the superficial layer containing carbides”
176: G.Maistro; S.A.Pérez-García; L.Nyborg; Y.Cao; “Thermal decomposition of nitrogen expanded austenite in 304L and 904L austenitic stainless steels”
183: Y.J.Lee; I.K.Kim; Y.B.Park; “Microstructures and Thermal Properties of Electroformed Invar Fine Metal Mask for OLED Deposition Processing”

105: C.Rotty; M.-L. Doche; A. Mandroyan; S. Monney; S. Lallemand; J.-Y. Hihn; “Roughness characterization procedures for electropolished brass”
172: L. Escalada; A. Gasco Owens; E. L. Dallison; G. Marcos; T. Czerwiec; S. P. Bruhl; S. N. Simison; “Corrosion and tribological performance of low-temperature carburized austenitic stainless steel. Role of the superficial layer containing carbides”
176: G. Maistro; S. A. Pérez-García; L. Nyborg; Y. Cao; “Thermal decomposition of nitrogen expanded austenite in 304L and 904L austenitic stainless steels”
183: Y. J. Lee; I. K. Kim; Y. B. Park; “Microstructures and Thermal Properties of Electroformed Invar Fine Metal Mask for OLED Deposition Processing”

16:00-16:20

Coffee Break

16:20-18:00

Poster session
184: F.Schiebel; M.Senn; F.Vallerani; M.Sebastiani; C.Eberl; “Residual stress measurement in metallic three-layered thin films”

186: B.Antoniszewski; M.Scendo; J.Trela; “Corrosion Properties of DLC Coating on Steel in Ringer Solution”

189: T.Tański; P.Jarka; W.Matsysiak; L.Krzemiński; B.Hajduk; M. Bilewicz; “Influence of technological conditions and content of nanoparticle reinforcing phases TiO₂ and Bi₂O₃ on optoelectronic properties and morphology of composite materials with a polymeric matrix”

201: T.Tański; W.Matsyiak; L.Krzemiński; P.Jarka; M. Bilewicz; “Analysis of the morphology, technological conditions and properties of SiO₂ nanowires obtained by the electrospinning method”

203: G.Marcos; A.Andrieux; J.Ghanbaja; T.Czerwiec; “Surface nitrogen content and properties of expanded austenite in 316L steel during a low-temperature plasma-assisted nitriding”

205: C.Zhang; S.D.Zhao, D.W.Zhang, C.C.Zhu; “Research of radial forging technology for planetary roller screw of high surface hardness”

212: L.Böhme, C.Godard, E.Kerscher; “Influence of engineered surfaces and microstructure on the fatigue limit of titanium”

215: O.Tkachuk, R.Proskurnyak, I.Pohrelyuk; “Thermo diffusion oxidation of implants from Ti-BaI-4V alloy”


223: A.Malachowska; M.Winnicki; M.Rutkowska-Gorzyczka; M.Korzeniowski; “Possibility of PC metalization with low pressure cold spray method.”

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CORROSION BEHAVIOUR OF SUPERHYDROPHOBIC
ALUMINUM SURFACES

L. Ruiz-de-Lara, R. Jagdheesh* and J.L. Ocaña

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Abstract

Manufacturing industries heavily depends on aluminum metal due to lightweight and good thermal properties. Aluminum (Al) is the most abundant metal elements in the crust. However, the low corrosion resistance limits the application in corrective environments. In this study, the electrochemical behaviour of superhydrophobic aluminum surfaces fabricated by nanosecond laser sources in a chloride environment was evaluated. Electrochemical behaviour was investigated using cyclic polarization curves and open circuit potential (OCP) measures. Cyclic polarization measurements were used to characterize the generalized and localized corrosion and OCP measurements are used to analyze the natural tendency of the material to be corroded. The superhydrophobic surfaces exhibited improvements in corrosion rate and polarization resistance due to an oxide layer formed during the laser treatment and the surface geometry itself.

Keywords: laser structuration; nanosecond; superhydrophobicity; corrosion; aluminum.

1. Introduction

In recent years, the generation of functional surfaces emulating natural structures has gained considerable interest due to their potential industrial applications. Among the most sought functional properties, the high degree of water repellence (superhydrophobicity) characteristic of the lotus (nelumbo nucifera) leaf has gained increasing interest for research due its anti-corrosion and low hydrodynamic friction properties [1]. Inspired by these water repellence and self cleaning properties of the lotus leaf, successful attempts have been made to change the surface wettability of different materials in two main different ways or a combination of both: (a) by modifying the surface chemistry by means of chemical coatings in order to reduce the surface free energy [2]; (b) by surface micromanipulation in order to generate micro/nano dual scale (hierarchical) surface roughness patterns [3]. Concerning the different methods for the achievement of the referred modification of wetting properties, different kinds of techniques like photolithography, nano casting, extrusion, placement of carbon nano tubes, etc., have been adopted with the common objective of creating a large-scale roughness in a variety of materials. However, these techniques suffer from weaknesses related to the high cost of projection masks and high development times in the different steps involved in creating the desired periodical roughness patterns [1].

At their turn, ultrafast laser sources can be used to create a variety of micro/nano structures in open environment in a reliable way. Normally, the superhydrophobic property can be realized through a controlled ablation patterning of the surface and subsequent application of different chemical coatings on the machined area in order to decrease the generally high surface energy of the initially machined surface in a classical "two-step” process. However, recent studies on laser micromachining have demonstrated their ability to fabricate micro/nano scale features with very limited distortion to the peripheral area [4, 5] and superhydrophobic effects were reported on ceramics by single-step (i.e. direct laser ablation) procedure by one of the authors [2]. Considering, on the other hand, that the application of an external coating material onto the laser structured surface is in general undesirable (especially in the case of metallic surfaces) as it manipulates the surface chemistry with resulting limited durability of the superhydrophobic behaviour, the seek for the capability of a one-step technique introducing the required modified wettability properties, especially in metallic substrates, seems to be a clear practical need.
Different authors ([2, 5]) have investigated thoroughly the application of short and ultrashort laser pulses, predominantly in the IR domain, to the ablation micromachining of different materials, including metals, concluding on the convenient use of lasers with pulses in the ps regime in order to avoid excess of material heat affected zones [6] and associate thermal effects. However, not much research has been published on the application of lasers with pulses in the ns regime and with emission in the VUV domain to the generation of surface micro/nanostuctures. Concerning the concrete field of the transformation of the wettability properties of different metallic surfaces, several interesting studies have been published on the direct applicability of lasers at different fluence levels for the generation of different degrees of hydrophobicity ([7,8]) and on the use of fs and ns pulses for the generation of multiscale surface features conceptually useful from the point of view of generation of hierarchical surfaces [9-13]. On the basis of these advantageous possibility, the work presented in this paper deals with the use of ns lasers with emission in the UV ($\lambda$=355 nm) to the controlled generation of different kinds of micro/nano features presumably leading to modified wettability properties (high degree of hydrophobicity) in typical metallic materials (Al being used as a representative sample). Concretely, ns laser pulses were applied on flat substrates of aluminum (Al) to fabricate micro pillars. The fabricated micro pillars have been studied with respect to the generation of high static contact angles (SCAs) and its corrosion resistance with respect to the wetting property.

2. Experimental

Flat aluminium (Al) sheets 100 $\mu$m thickness and negligible roughness were laser treated over 5 mm x 5 mm areas with the aid of a fully automated laser micromachining workstation incorporating a Spectra-Physics Pulseo® 355-20 DPSS laser with average power in excess of 20 W at $\lambda$=355 nm and 100 kHz repetition rate at the UPM Laser Centre. The laser beam was guided over the samples surface by an optical system that included six mirrors, a beam expander, a digital scanner and a lens with 250 mm focal length. The experiments were performed at fixed pulse duration of about 30 ns. The laser beam has Gaussian power density profile and the machining was performed in atmospheric conditions with an apparent spot size of 15 $\mu$m. Micro pillars were produced at a power 300 mW. Micropillars were separated by a distance in a range of 10-25 $\mu$m. The distance is hereafter termed as pitch “P” for future reference in the text. The detailed laser processing conditions are listed in Table 1. The laser machined surface patterns were analyzed with scanning electron microscope (SEM) equipped with Energy dispersive X-ray spectroscopy (HITACHI, Model S-3000N®) and confocal laser scanning microscope (CLSM, LEICA DCM 3D®) to evaluate the geometry of the induced patterns. The hydrophobicity/water repellence of the samples was studied by measuring the static contact angle using the sessile drop technique, with a video-based optical contact angle measuring device (OCA 15 Plus® from Data Physics Instruments). 8 $\mu$L droplets of distilled deionized water were dispensed on the laser-machined surface structures under atmospheric conditions, and the static contact angle was calculated by analyzing droplet images recorded just after the drop deposition.

Table 1: Laser Processing parameters used for the generation of the described superhydrophobic patterns.

<table>
<thead>
<tr>
<th>Laser Power, $P_l$ (mW)</th>
<th>300</th>
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<tbody>
<tr>
<td>Spot diameter, $D$ ($\mu$m)</td>
<td>15</td>
</tr>
<tr>
<td>Fluence, $F$ (J/cm²)</td>
<td>1.6</td>
</tr>
<tr>
<td>Repetition Rate, $R$ (kHz)</td>
<td>100</td>
</tr>
<tr>
<td>Scan Speed,$v$ (mm/s)</td>
<td>40</td>
</tr>
<tr>
<td>Pitch, $P$ ($\mu$m)</td>
<td>10, 15, 20, 25</td>
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The corrosion behavior in a corrosive solution medium has been studied by means of cyclic polarization techniques [14] using a three electrode cell, being the sample under test the working electrode (1 cm²), a carbon bar as the counter electrode and a saturated sodium chloride 3M as the reference electrode. A NaCl 0.5 M aqueous solution was employed as the electrolyte. Open circuit potential (OCP) measurements were done during 3600 s in order to show the natural tendency of the material to be corroded. Electrochemical corrosion measurements were performed on an electrochemical workstation (Metrohm Autolab AUT 85349) with a computer-controlled potentiostat/galvanostat connected to an electrochemical cell. The polarization curves were performed at a scan rate of 0.5 mV/s and all the measurements were conducted at room temperature. An examination of corroded surfaces with scanning electron microscopy was done using the referred Hitachi S-3000N SEM.
3. Results

3.1. Microstructure and modified wettability character

Figure 1 shows the confocal 3D image of the micro pillars fabricated with four different values of P (10, 15, 20 and 25 µm) with 300 mW laser average power. The micro pillars fabricated with P=10 µm and P=15 µm resulted into scattered peak instead of peaks at regular intervals. Heavy deposition of recast material on the top surface of the micro pillars is observed in these samples, whereas the micro pillars fabricated with P=20 µm and P=25 µm have debris deposition over a broader circumference area. The corresponding SEM images (figure 2) indicate the melt formation during the laser ablation. It is clear that the molten material solidifies along the direction of the laser motion. For all the four values of P, the channels are opened in one direction and closed on the perpendicular direction. This is caused by the ejection of the melt formed during the laser processing. In addition to the heavy piling up of recast material on the top edges of micro pillars, spherical shaped resolidified metal vapour structures are also found. This may correspond to the self-cooled metal vapour from the plasma generated by laser ablation.

![Image of CLSM 3D images of the micro-pillars fabricated with P=300 mW, v=40 mm/s and f =100 kHz for four different values of P: A) 10 µm; B) 15 µm; C) 20 µm; D) 25 µm.]

![Image of SEM image of the micro-pillars fabricated with P=300 mW, v=40 mm/s and f =100 kHz for four different values of P: A) 10 µm; B) 15 µm; C) 20 µm; D) 25 µm.]

In this kind of structures, the piling of the recast is observed to form a µ-cell or closed-packet-like structure on the top of the micro pillars as shown in figure 2. In this case, the µ-cell structure is separated by 10-15 µm channels. Therefore, the laser patterned aluminium surface has two different patterns such as micro pillars and µ-cell, thus, reducing the fraction of solid area in contact with the water droplets. This fact provides an important advantage as for as water repellence is concerned. The closed micro channels act as micro packets and...
can hold small volumes of air trapped inside the pockets what, in turn, can be used to prevent the water to flush out the air inside the narrow micro channels.

The effect of the generated micro patterns on the wetting properties of the Al samples was evaluated by static sessile drop contact angle measurements, using a droplet size of 8 μL. The plain Al surface recorded a SCA value of 85 ±3°. The entire laser patterned surface was highly hydrophilic within minutes of processing but the SCA measurements performed after 24 hours and after different successive time periods invariably exhibited the hydrophobic character, what provides a strong argument about the stability of the generated structures. The SCA measurements shows increase in SCA values with respect to P and starts declination beyond P=20 μm for micropillar structures. For the cases where the water droplets are unable to be dispensed on the surface from the micro-syringe, the normal sessile droplet technique is not applicable. The SCA of these kind of surfaces is considered to be 180° [9,15] or ultrahydrophobic surface. The low values of SCA for the sample processed with P=10 μm may be due to the lack of formation of the μ-cell structure. Samples with micro pillar geometry exhibited ultrahydrophobicity for the samples processed with P=15 μm and 20 μm. The sample processed with P=25 μm recorded a SCA of 160°. When the droplet is dispensed on the laser patterned surface, the water droplet has contact only with the top edge of the μ-walls due to the small volume of air trapped in the μ-cell structures. The air bubbles act as a cushion for the water droplet, thus avoiding the contact with the bottom surface of the μ-cell structures. The contact line composed of solid-liquid-air has been explained by the Cassie Baxter model [6].

3.2. Corrosion behavior

First of all, open circuit potential (OCP) measurements were carried out. It was observed that, in general terms, the higher (less negative) OCP a material has, the harder to corrode it will be. The OCP values are shown in the Table 2, and although the results are very similar, the best situation is for the material treated with 20 μm pitch, coinciding with the best hydrophobic performance, this meaning that the superhydrophobic character can be initially considered as a property preventing the corrosion.

### Table 2. OCP measurements and contact angle.

<table>
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<tr>
<th>OCP (V)</th>
<th>Contact angle (°)</th>
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<tr>
<td>Bare aluminum</td>
<td>-0.754</td>
</tr>
<tr>
<td>P=15 μm</td>
<td>-0.704</td>
</tr>
<tr>
<td>P=20 μm</td>
<td>-0.695</td>
</tr>
<tr>
<td>P=25 μm</td>
<td>-0.704</td>
</tr>
</tbody>
</table>

Additionally, the corrosion resistance of the generated aluminum superhydrophobic surfaces was evaluated by cyclic polarization in NaCl 0.5 M aqueous solution, the results being displayed in figure 3. In this kind of tests, a lower corrosion current density implies a lower corrosion dynamic rate [17] and better anti-corrosion performance [18]. Through the available electrochemical analysis software, different parameters were extracted from cyclic polarization curves and are shown in Table 3. Corrosion potential (E_{corr}) and corrosion current density (I_{corr}) for different specimens were obtained using the Tafel method [19] by extrapolating the anodic and cathodic slopes.

While the corrosion potential (E_{corr}) is related with the active zone where the material is been corroded, the corrosion current density is linked with the corrosion resistance. Therefore attending to the results in Table 3, an superhydrophobic character implies a better anti-corrosion behavior than that corresponding to bare material. And, considering only the superhydrophobic surfaces, the best superhydrophobic performance, micropillars fabricated with 20 μm pitch has the lower corrosion current density (i.e. a higher resistance to corrosion).

### Table 3. Parameters obtained from cyclic polarization plots in Fig.3.

<table>
<thead>
<tr>
<th>E_{corr} (V)</th>
<th>I_{corr} (nAcm^{-2})</th>
<th>Corrosion Rate (μm/year)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bare Aluminum</td>
<td>-0.753</td>
<td>215.3</td>
</tr>
<tr>
<td>15 HD</td>
<td>-0.781</td>
<td>14.4</td>
</tr>
<tr>
<td>20 HD</td>
<td>-0.810</td>
<td>11.3</td>
</tr>
<tr>
<td>25 HD</td>
<td>-0.747</td>
<td>82.4</td>
</tr>
</tbody>
</table>
Fig. 3. Cyclic polarization curves of aluminum, (a) bare material; (b) micropillars fabricated with P=15 µm; (c) micropillars fabricated with P=20 µm; (d) micropillars fabricated with P=25 µm.

Trying to find a physical explanation to the reported results, the µ-cell structures created with this kind of laser fabrication are considered the main factor responsible for the observed behavior. Because of the air bubbles kept trapped in the generated microstructure, the NaCl solution and the corrosive ions are presumably maintained away from the direct contact with the bottom surface, this being a major factor for the improvement of the corrosion resistance [17, 20, 1]. Because of this situation, the corrosion current density is reduced by 95% when the laser fabrication is performed with a pitch of 20 µm compared with the bare material, while respective reductions is 93% and 62% compared to the bare material for pitch values of 15 and 25 µm are obtained. The same tendency for the corrosion rate is obtained: respective reductions of 91%, 93% and 51% compared with the bare material are obtained for 15, 20 and 25 µm pitches.

In order to have a better comprehension of how the superhydrophobic material is affected by NaCl aqueous solution, a study using a scanning electron microscope (SEM) was done. In the figure 4 different images of the corroded surfaces at different magnifications are shown.

Fig. 4. SEM images of corroded aluminum superhydrophobic surfaces, (a) fabricated with 15 µm pitch; (b) fabricated with 20 µm pitch; (c) fabricated with 25 µm pitch. The number 1 indicates a general view, the number 2 a detail of a part of surface that is starting to be corroded and the number 3 shows a pit.

The principal fact observed in these images is that the surface fabricated with a pitch of 25 µm has been more affected by the corrosion (more pits) (Fig. 4.c1) in comparison with the other two parameters of fabrication (Fig. 4.a1 and 4.b1). Such differences are related to the pitting potential (the least positive potential at which pits
can form, Epit) and the corrosion rate. The pitting potential is obtained from cyclic polarization curves, being the value of potential from which corrosion current density begins to increase rapidly in the anodic branch. The pitting potential for the different values of pitch (15, 20 and 25 µm) is -0.730 V, -0.626 V and -0.783 V respectively. The highest value (less negative), for which it is more difficult to initiate a pit, coincides for the best corrosion resistance performance, 20 and 15 µm of pitch, showing less pits than the other situation (Fig. 4.b1 and c1). The superhydrophobic surface fabricated with 25 µm of pitch exhibits more pits (Fig. 4.c3) than the other situations (Fig. 4.a3 and 4.b3) because it has the lowest pitting potential and the surface is very affected by the NaCl aqueous solution.

4. Conclusions

The present investigation has shown a direct way to create superhydrophobic surfaces with ns laser in aluminum, a method that can be extended to other materials or/and alloys. Deepening in others characteristics of this kind of surfaces, the corrosion resistance were analyzed with cyclic polarization curves and scanning electron microscopy. The corrosion resistance was observed to be improved, presumably as a consequence of the due to the bubbles of air trapped in the hierarchical micro-nanostructures generated around micropillars avoiding the penetration of corrosive species (such as Cl-) to contact with the aluminum surface. The better anti-corrosion behavior matches with the highest superhydrophobic performance, linking the superhydrophobic character with the anticorrosion behavior and the definition of optimized microfabrication pitches being perfectly achievable.

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