PROGRAMMA INTERREG V A Italia – Austria 2014-2020 Progetto ICAP ITAT1010

### "Innovazione tramite applicazioni combinate delle tecnologie al plasma" "Innovation durch kombinierte Anwendungen von Plasmatechnologien"











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### Activation of surfaces: a comparison between vacuum and atmospheric plasma performances

Giuseppe Da Cortà



## Activities of ICAP Project :

- Identification of the needs of SME in the program area;
- Performing experiments with different plasma technologies
- Dissemination, information and promotion of studies and results.



## Economical features of Belluno industrial districts

- The most important manufacturing field is the optical industry.
- The global trade of the optical business is 4 millions pcs/day
- Italy is a leading country in the optical business



## Equipment



#### Vacuum Plasma :

- Pico-PC
- HF generator : 40 kHz, 200W









Atmospheric plasma: Frequency 20 kHz 20 kHz Power: 300 W Movement device: x,y,z robot

# Surface energy calculation for optimization of processes

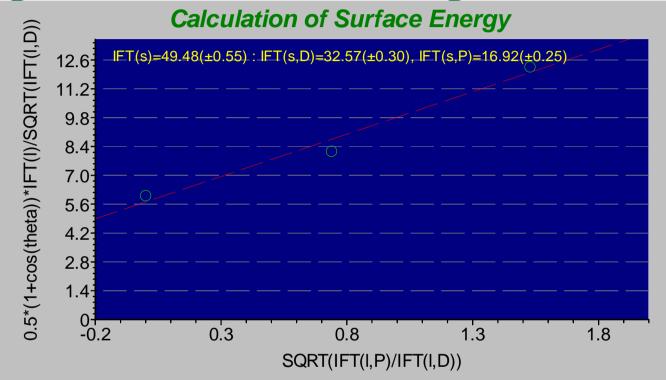
- Substances with low surface energy are able to wet those with high surface energy, but not vice versa.
- The surface energy of the applied liquid must be lower than that of the substrate, otherwise molecules cannot find connection points to which they can cling.

The surface energy of a surface is increased by activation: this process ensures the creation of anchorage points for the applied liquid.

## Surface energy calculation for optimization of the processes



## Surface characterization and optimization of the processes



#### **Optical measurements of contact angle:**

- Sessile Drop Method
- Test Liquids: Water, Ehylene glycol, Diiodomethane (parameters by Ström et alt.)
- Drop shape and contact angle calculation with Young Laplace equation.
- Surface energy calculation with OWRK (OWENS, WENDT, RABEL and KAELBLE) equation.





Atmospheric plasma	Low pressure plasma
Cleaning/ activation/coating	Cleaning/ activation/etching/coating
Suitable for 2D components	Suitable for 3D components
Integrable in automated plants	Suitable for bulk materials
Continuous in-line processes	Batch processes
High speed treatments	Optimization of required parameters
Treatments are unproblematic on the most sensitive substrates such as wood, textiles, polymers.	Low pressure conditions required
Economical and low-maintenance	Higher investment
Low energy consumption	High energy demand (pumps)



Given that the main factor to obtain the best result of a surface treatment is its preparation, this project was focused on the pre-treatments of surfaces.

Traditional pre-treatments are:

- flame treatments,
- wet washes with detergents or solvents,
- mechanical treatments such as sanding or grinding
- use of primers or electrochemical treatments.



Surface preparation

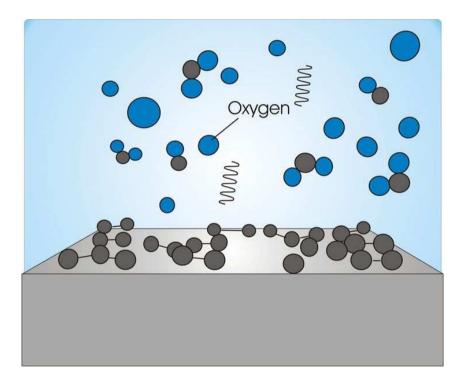
Plasma allows a perfect micro-cleaning of surfaces through the plasma reactions:

- oxidation,
- surface bombardment by electrons,
- u.v radiation
- surface ablation.





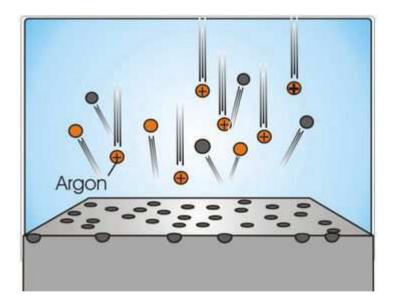
By processing Oxygen, organic contaminants are removed: this reactive gas, together with the energy and radiations of plasma, breaks the organic chains forming  $H_2Oe$  $CO_2$ , which evaporate.







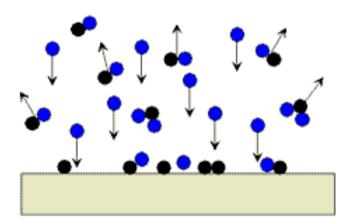
It is made by processing noble gases, mainly Ar which is the least expensive. The action is purely mechanical, i.e. the particles impact the surface of the substrate by undermining molecules, which are crushed by the plasma and removed.



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This treatment acts on the surface by implanting atoms and / or functional groups that modify its properties by varying the affinity of the surface itself: e.g. functional groups containing Oxygen increase the hydrophilicity, functional groups containing halogens increase the affinity to fats.







The final effect of a treatment is the result of a dynamic balance:

- **Time** determines the balance between the particles that are implanted and those that are removed, since they are on the surface and in a state of stability lower than that of the material itself.
- **Pressure** determines the quantity of reagents in the chamber: by increasing it, the number of impacts increases but the energy of the particles gets lower, due to the decrease of the free average path;
- **Power** fragments the precursors and determines the acceleration of the particles, that is the energy of the impacts.

The best performing parameters can be determined only experimentally.

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## Outputs of SMEs needs: Optimization of fabric gluing by microcleaning and activation by using vacuum and atmospheric plasma.







### Substrates:

- Steel
- Aluminium
- Acetate
- Galvanic coated metal
- Varnished metal
- Metal coated with Teflon





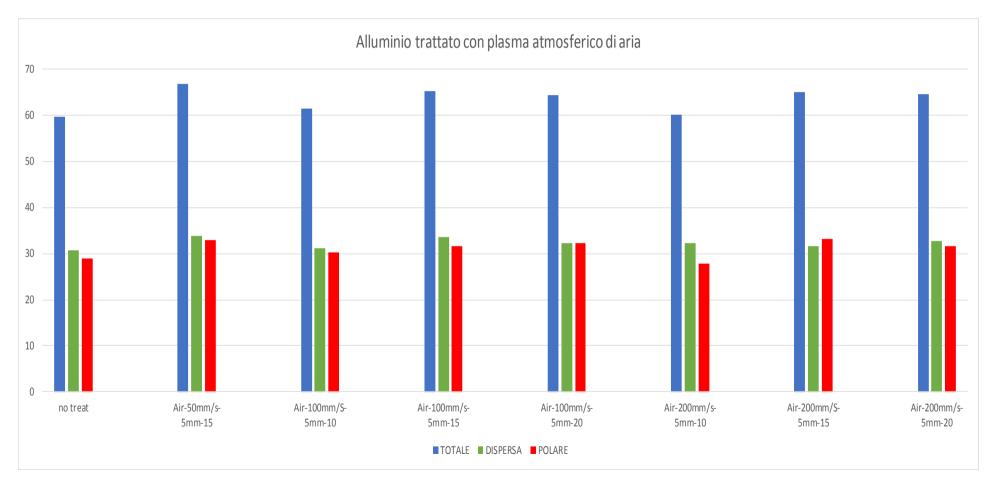


 This presentation will focus on the activation for the gluing of textiles on steel and alluminium hard cases





#### Atmospheric plasma





## Al plates treated with Air atmospheric plasma

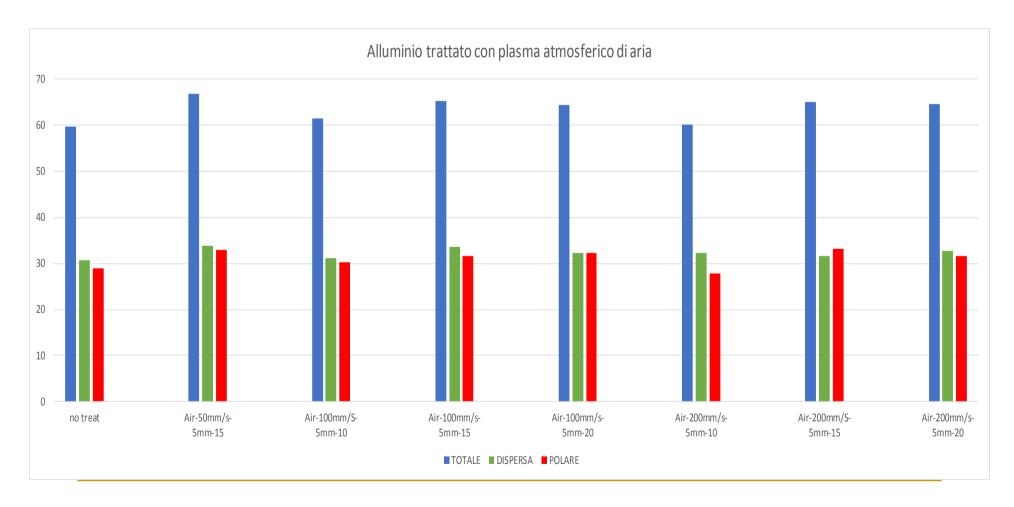
- Distance between torch and substrate: 5mm
- [mm/sec]: speed of the plasma device
- 10 15 20 : number of revolutions to fill the rectangle i.e. distance between two lines
- In the case of Al plates, treatments are not effective

Parameters	total	disp	polar
untreat	59,7	30,6	32,9
50mm/sec - 15	66,8	33,8	32,9
100mm/sec - 10	61,4	31,2	30,2
100mm/sec - 15	65,1	33,5	31,5
100mm/sec - 20	64,3	32,3	27,9
200mm/sec - 10	60,1	32,2	27,9
200mm/sec - 15	64,9	31,6	33,1
200mm/sec - 20	64,5	32,8	31,6



## Aluminium

#### Vacuum plasma







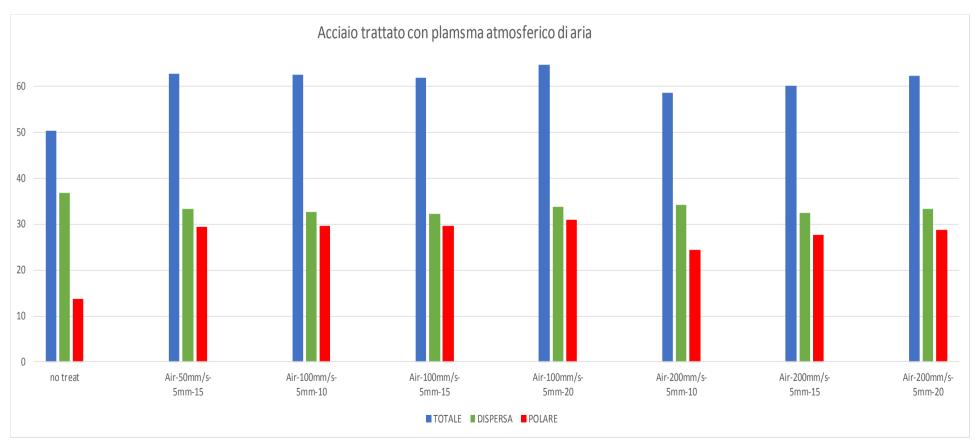
Also the vacuum plasma treatment is not effective on Al plates

gas	press	power	tot	disp	pol
untre	untreated		59,7	30,6	29
O <sub>2</sub>	0,3	90%	60,5	30,9	29,6
	0,6	90%	64,3	29,7	34,6
	0,3	70%	61,8	33	28,8
Ar	0,3	90%	62,7	32,6	30,1
	0,6	90%	62,6	33	30,5
	0,3	70%	63,8	33,5	30,3
Air	0,3	90%	65,3	33,2	31,1
	0,6	90%	62,6	28,8	33,7
	0,3	70%	61,6	29,7	31,8



### Steel

#### Atmospheric plasma





## Steel plates treated with Air atmospheric plasma

In the case of steel plates, treatment is effective:

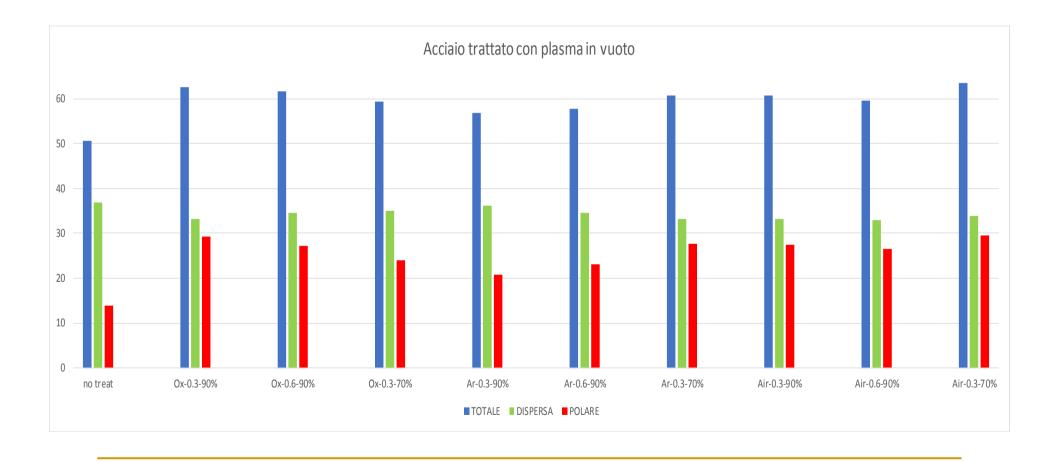
best performing parameters are highlighted in red

Parameters	total	disp	polar
untreat	50,3	36,8	13,7
50mm/sec - 15	62,7	33,3	29,3
100mm/sec - 10	62,4	32,7	29,7
100mm/sec - 15	61,8	32,3	29,6
100mm/sec - 20	64,6	33,7	30,8
200mm/sec - 10	58,5	34,2	24,3
200mm/sec - 15	60	32,4	27,6
200mm/sec - 20	62,2	33,4	28,8



Steel

Vacuum plasma



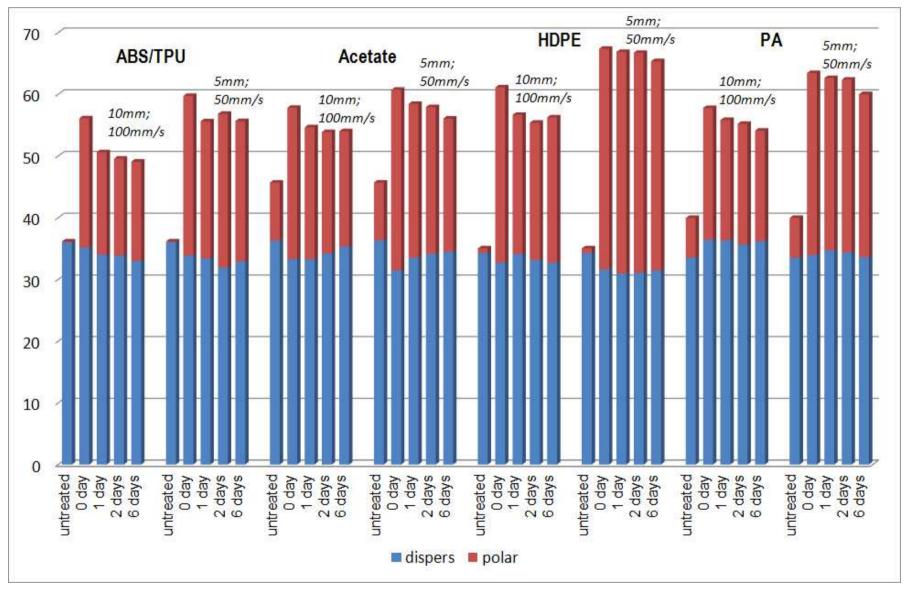


### Steel plates treated with vacuum plasma:

vacuum plasma treatments are effective as well.

gas	press	power	tot	disp	pol
untre	untreated		50,7	36,8	13,8
O <sub>2</sub>	0,3	90%	62,5	33,2	29,2
	0,6	90%	61,6	34,6	27,3
	0,3	70%	59,3	35,1	24
Ar	0,3	90%	56,9	36,1	20,8
	0,6	90%	57,8	34,5	23,2
	0,3	70%	60,8	33,1	27,6
Air	0,3	90%	60,8	33,3	27,4
	0,6	90%	59,7	33	26,6
	0,3	70%	63,4	33,8	29,6

## **Durability tests on plastics (W3C)**





## Durability on steel

	adhesive	untreated	$O_2 \div C1$	$O_2 \div C2$	$O_2 \div C3$
Tot.	33	45	63	67	66
3 days			35	58	43
Disp.	31	32	32	31	30
3 days			31	32	33
Polar	2	12	31	36	36
3 days			4	26	10

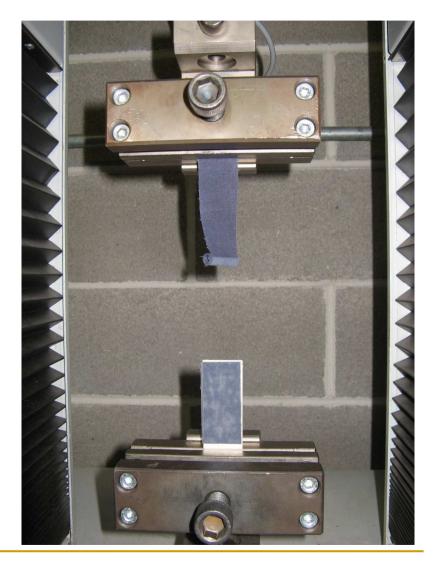
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# Simulation of the processes and performance evaluation

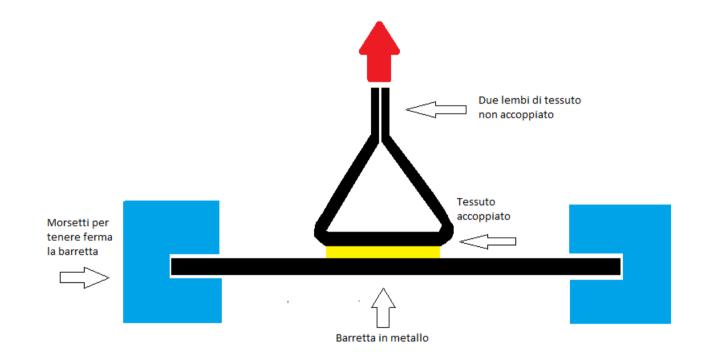
#### Interreg Italia-Österreich EURPEAN UNDER TEAR Test with tension machine

 Results obtained by placing the fabric sample vertically between the plates would not have been acceptable due to delamination.





## Test with tension machine



#### Interreg Italia-Österreich urgen Region Der Linder in the tension machine

 The plate was placed horizontally, blocking it with two clamps and 2 edges of the textile have been gripped: results are more realistic



#### Interreg Italia-Österreich EUROPEAN UNICH IGAR Performance evaluation

- The force needed to tear the thermoadhesive fabric from the metal plate was measured.
- With both technologies, there is a surface activation.
- Vacuum plasma is more effective

Plasma	Parameters	[N] <sub>max</sub>
Untreat.		62,35
Vacuum	0,3 [mbar]	82,52
10'	0,6 [mbar]	82,32
Atmosph.	100 [mm/sec ]	67,62
5 mm.	100 [mm/sec ]	74,84

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## Thank you for your attention!









