Biomimetic sol-gel-coatings: Structuring by femtosecond Laser

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Motivation

The sol-gel techniques are relatively low-cost, the reaction conditions are mild, and more-over the composition and structure of coatings can be easily controlled.

Sol-gel processing of alumina provides films with different microstructure [1-3]. Zhang et al [1] report that the wear life of sol-gel generated Al_2O_3 coatings can be very short, if the coating has no appropriate microstructure. They observe fine abrasive particles can form with ongoing wear. Some sol-gel alumina coatings showed slight plastic deformation according Zhang et al. [1]. Such coatings showed a longer wear life.

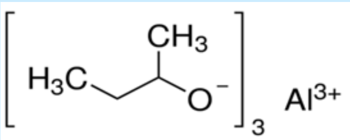
It is known, that surface topography plays an essential role in tribology. Laser impact is one possible method to create periodic surface topographies. Kunz et al. [2] describe laser-induced periodic surface structuring (LIPSS) gained rapidly increased interest in recent years. Not only wear behaviour, but wettability, optical properties or implementation of functional features to generate smart effects can be reached by reliable surface structuring of materials

Experimental methods

Sol-gel coatings were deposited by dip coating on stainless steel 1.4403 (X5 CrNiMo19-11) samples.

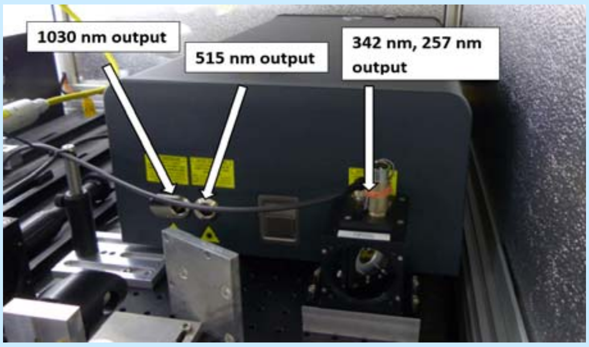
As precursor a solution of aluminium-tri-sec-butoxide $Al[OCH(CH_3)C_2H_5]_3$ (99,9%, Sigma-Aldrich) in methoxyethanol (99,8%, Sigma-Aldrich) was selected.

Following drying, the samples were heat treated in vacuum oven (0.002-0.008 mbar). Two different top temperatures were chosen, 900°C or 1100°C.



Alumina precursor $Al[OCH(CH_3)C_2H_5]_3$ for sol-gel synthesis of ceramic layers in stainless steel 1.4403

Laser treatment were used for generating the surface structure. The femtosecond laser Pharos 15-1000-PP of the company Light Conversion, Lithuania is a single-unit integrated laser system combining millijoule pulse energies and high average power.



Pharo laser system at the Laser institute Mittweida, Company Light Conversion

No.	Precursor	Top temperature	Laser treatment
1	$Al[OCH(CH_3)C_2H_5]_3$	900 °C	/
2	$Al[OCH(CH_3)C_2H_5]_3$	1100 °C	/
3	$Al[OCH(CH_3)C_2H_5]_3$	900 °C	P_{AV}: 1.62 W; E_P: 5.8 µJ
4	$Al[OCH(CH_3)C_2H_5]_3$	1100 °C	P_{AV}: 1.62 W; E_P: 5.8 µJ
5	$Al[OCH(CH_3)C_2H_5]_3$	900 °	P_{AV}: 9.80 W; E_P: 49 µJ
6	$Al[OCH(CH_3)C_2H_5]_3$	1100 °C	P_{AV}: 9.80 W; E_P: 49 µJ

P_{AV}: laser power; E_P: pulse energy

For laser structuring of the coated surface, pulse duration was 220 fs, repetition rate 220 Hz and laser beam radius 11.5 µm in focus. Two different surface structures were generated. A laser power P_{AV} of 1.62 W and a pulse energy E_p of 5.8 µJ were used to generate small dimpling. Bigger dimples were generated by a laser power P_{AV} of 9.80 W and a pulse energy E_p of 49 µJ.

References

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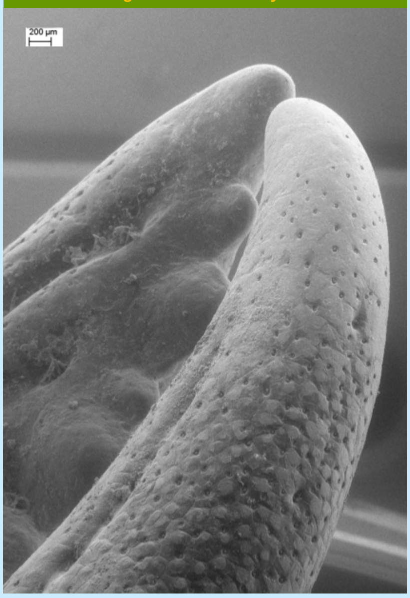
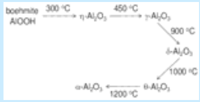
Crab shovel:

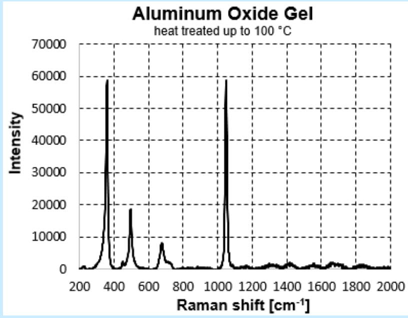
Resistance against sand in salty environment!

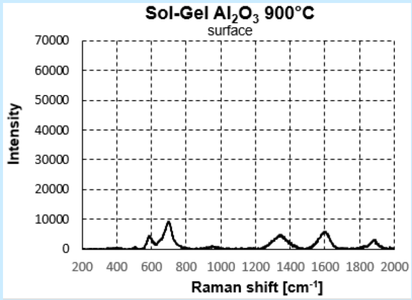


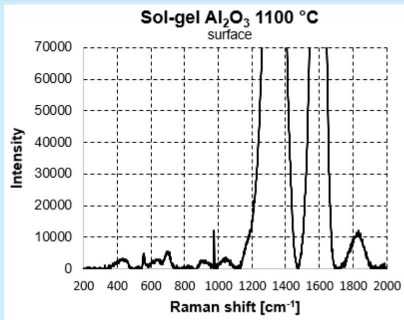
Results

Raman spectroscopy



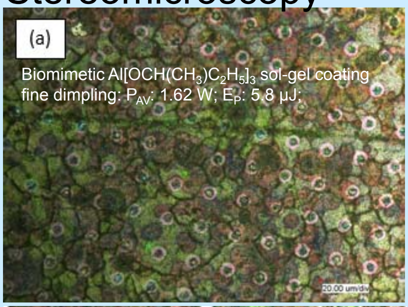


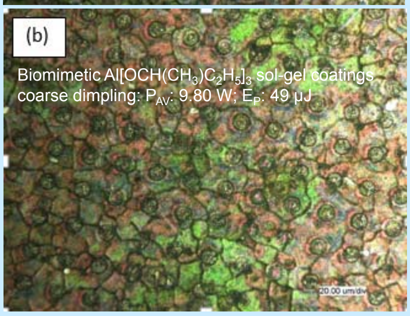




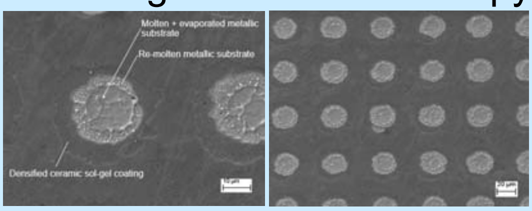
	Raman peak	Bonding	Reference
Boehmite (AlOOH) γ-Al_2O_3	420 cm^{-1}	Al-O vibration	[4]
	520 cm^{-1}	Al-O vibration	[4]
	620 cm^{-1}	Al-O vibration	[4]
	740 cm^{-1}	Al-O vibration	[4]
	980 cm^{-1}	bending vibration of OH	[4]
	1020 cm^{-1}	bending vibration of OH	[4]
	1060 cm^{-1}	bending vibration of OH	
Al_2O_3 (tetragonal) γ-Al_2O_3 400-900°C	460 cm^{-1}		[5]
	1196 cm^{-1}		[5]
	1260 cm^{-1}		[5]
Corundum α-Al_2O_3 (hexagonal) 1000-1100°C	1360 cm^{-1}		[5]
	1390 cm^{-1}		[5]
α-Fe_2O_3	227 cm^{-1}		[6]
	294 cm^{-1}		[6]
	412 cm^{-1}		[6]
	500 cm^{-1}		[6]
	612 cm^{-1}		[6]
	670 cm^{-1}		[6]
	1324 cm^{-1}		[6]
γ-Fe_2O_3	1423 cm^{-1}		
Fe_3O_4	670 cm^{-1}		[6]
FeOOH γ und α	1350 cm^{-1}		
	330-332 cm^{-1}	Fe-O stretching vibration	[7]
Hydrated chlorides	1640 cm^{-1}		[7]
Cr_2O_3	530 cm^{-1}		[7]
Cr(III) und Cr(IV) mixed oxides	860-870 cm^{-1}		[7]
CrO_3	950-970 cm^{-1}		[7]

Stereomicroscopy





Scanning electron microscopy



Conclusions

- Successful tests of biomimetic laser dimpling of Al_2O_3 sol-gel coating according the natural model of "Crab shovel".
- Oxide interlayer increases with heat treatment temperature intensively.
- Laser impact effects different heat treated zones with evaporation, melting and densification/ sintering of metallic or ceramic materials.