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Microscopic and spectroscopic studies of epoxy-steel interfaces

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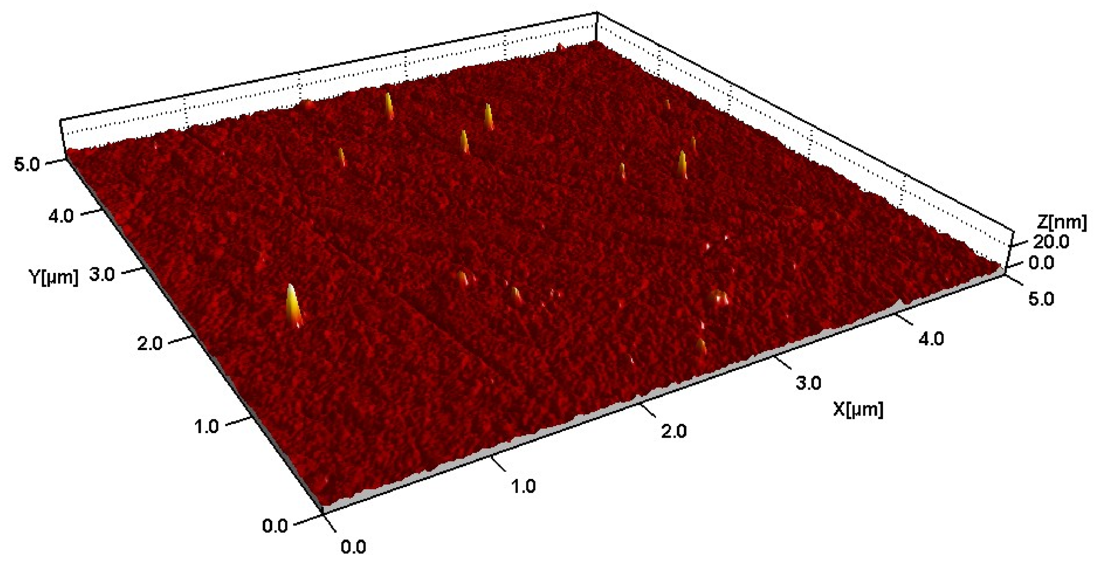
Introduction & Experimental

Deformable sandwich sheet materials produced as raw materials and formed afterwards to complex geometries require a good bonding to guarantee a stable shear transfer between the single layers. However, due to the deformation, failures by delamination often occur between the epoxy and the metal. The aim of our work is to understand the basic effect of the interfaces' bonding behavior in selected preparation steps of the system. These effects have been investigated by means of Atomic Force Microscopy (AFM) , operating in ambient air and X-ray Photoelectron Spectroscopy (XPS), operating under ultrahigh vacuum conditions.

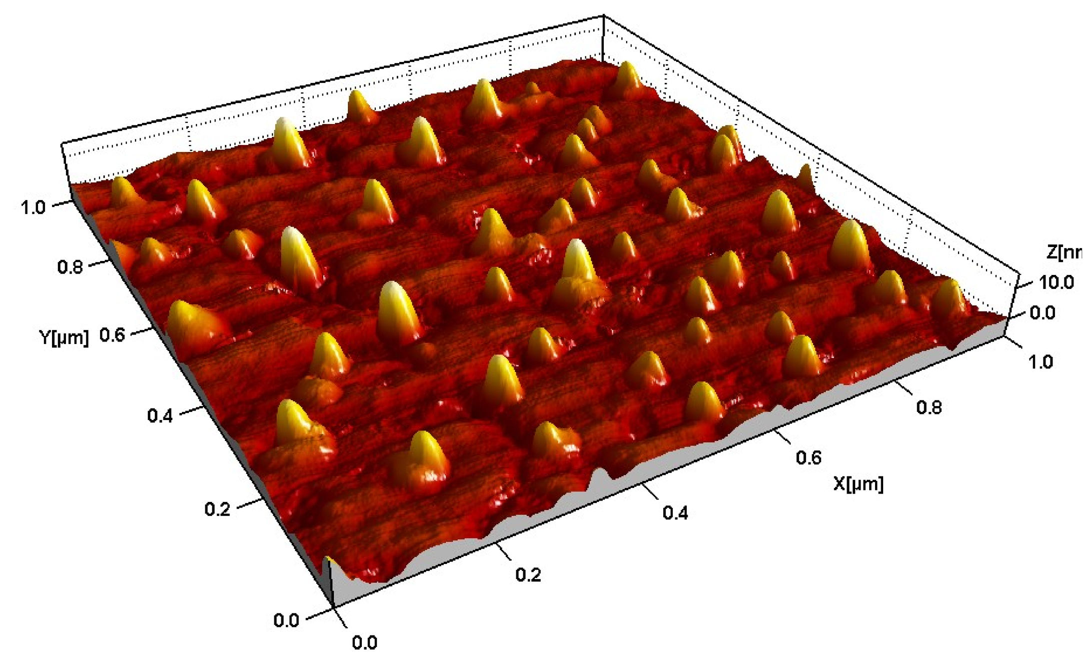
Preparation:
- **1st step:** The pure steel surface was manually cleaned with acetone and isopropanol, as well as sputtered with Ar^+ ions.
- **2nd step:** The sample from step one was extracted from vacuum and exposed to ambient air for 2 min.
- **3rd step:** The sample from step two is covered with an epoxy film by spin coating.

AFM 1st step



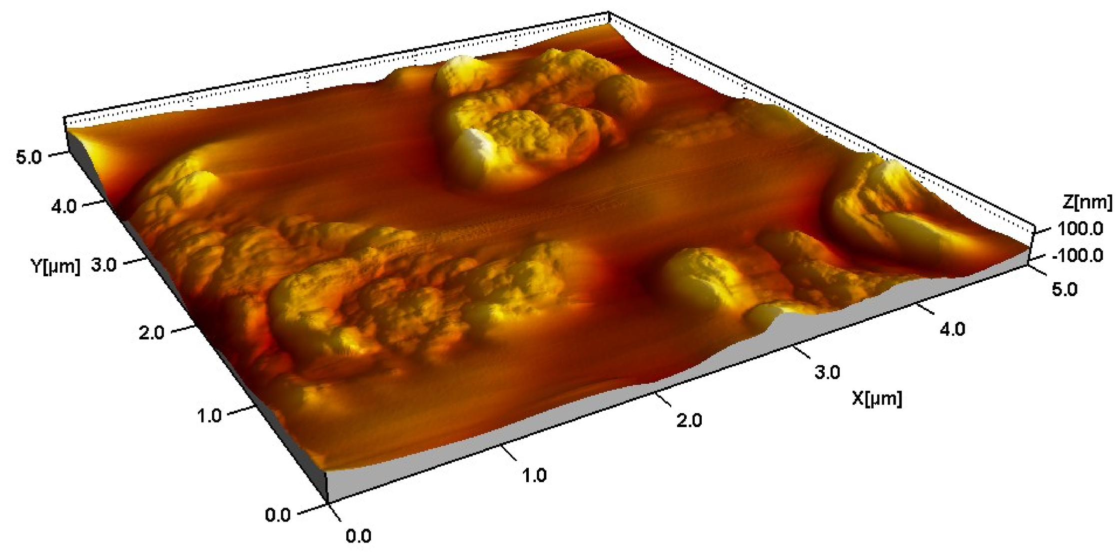
smooth surface
distinct structures
(probably aerosols)

AFM 2nd step



aerosol particles
(arising from agglomeration)
within the atmosphere)

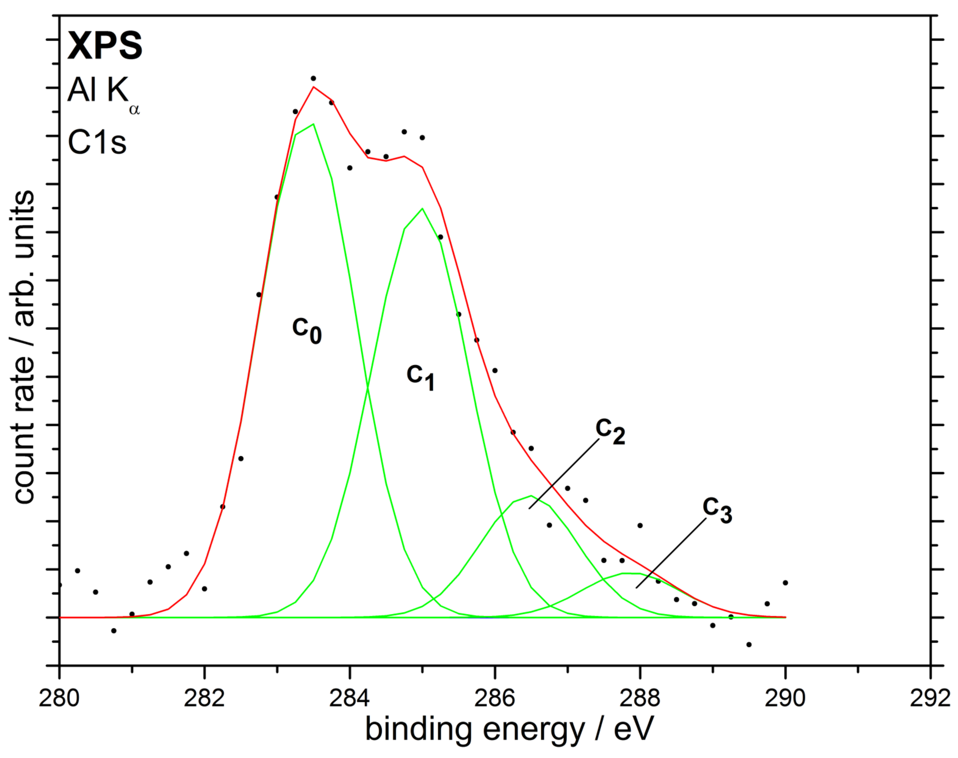
AFM 3rd step



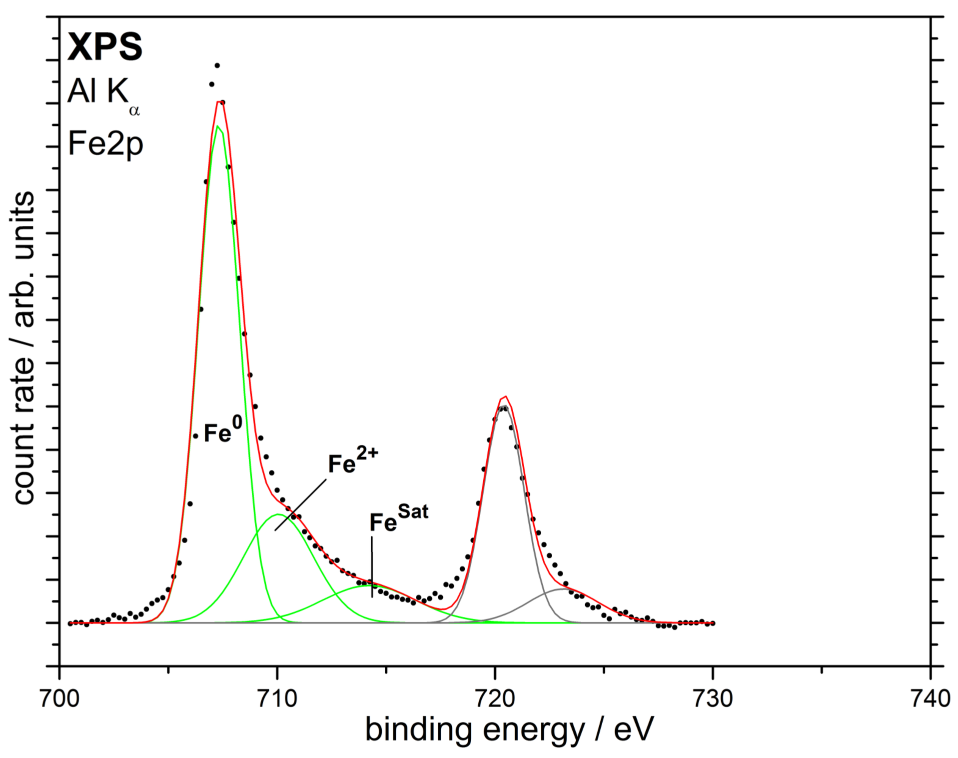
two different domains
regions with a comparatively smooth surface
regions which exhibit noticeable aggregation of epoxy

	1st step	2nd step	3rd step
RMS roughness:	1.5 nm	3.7 nm	34 nm
avg. diameter:	50-150 nm	129 nm	-
mean height:	30 nm	28 nm	-

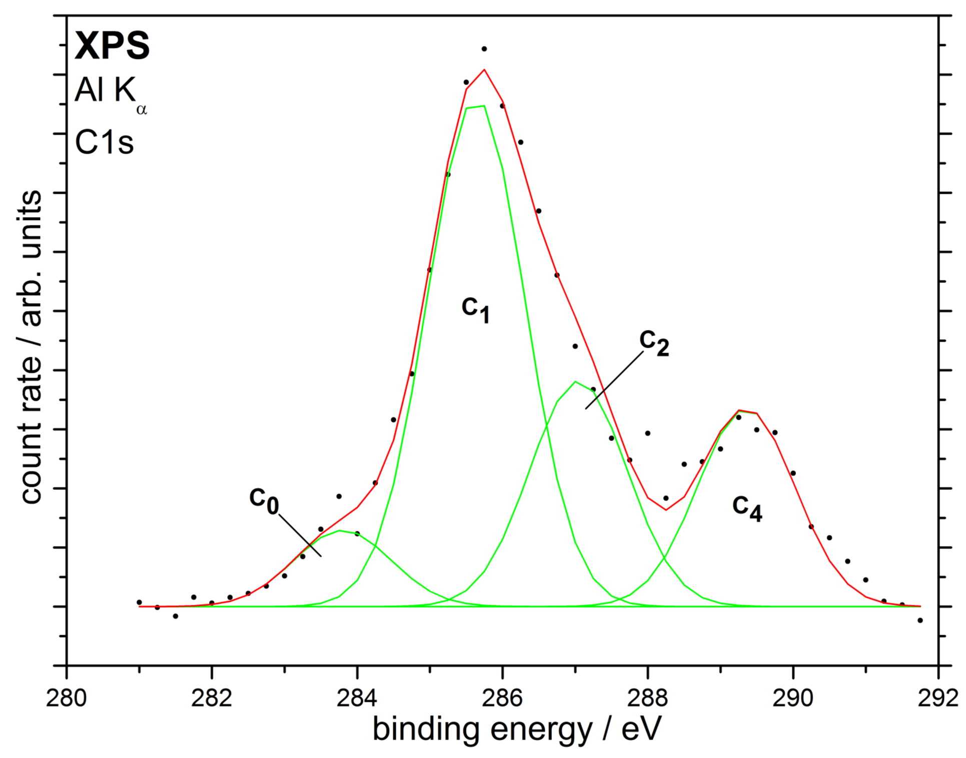
XPS 1st step



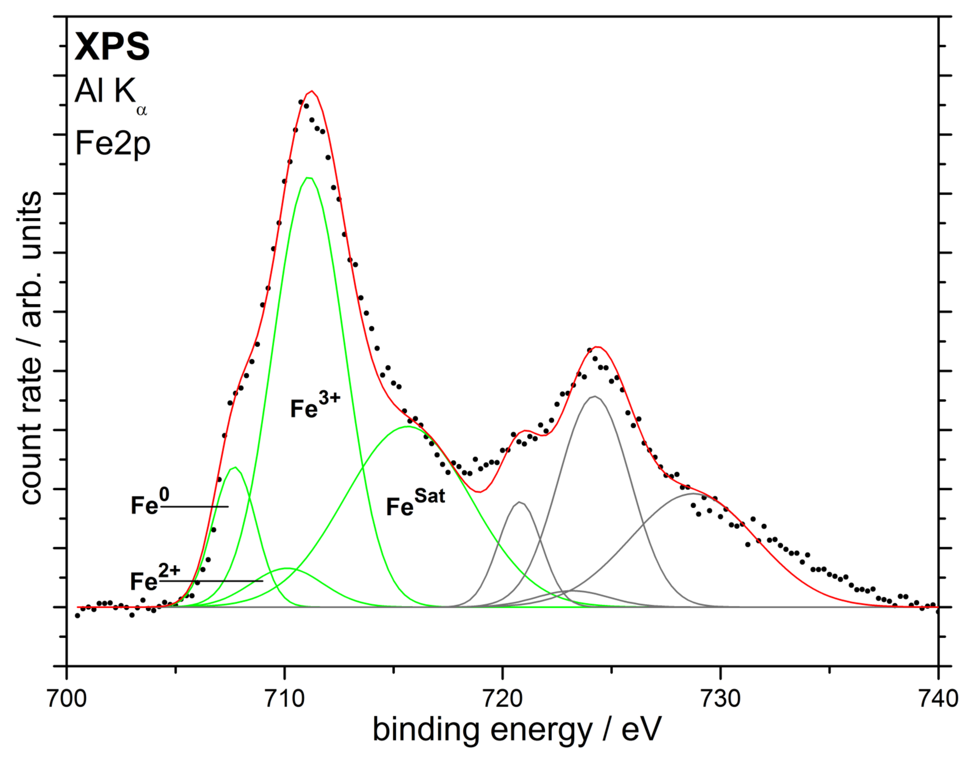
Orbital	Peak	rel. int.	Assignment
C1s	C_0	46.3 %	carbide
	C_1	38.1 %	C-C / C-H
	C_2	11.4 %	C-O
	C_3	4.2 %	C=O
Fe2p	I	72.8 %	Fe^0
	II	27.2 %	Fe^{2+}
	III	-	-



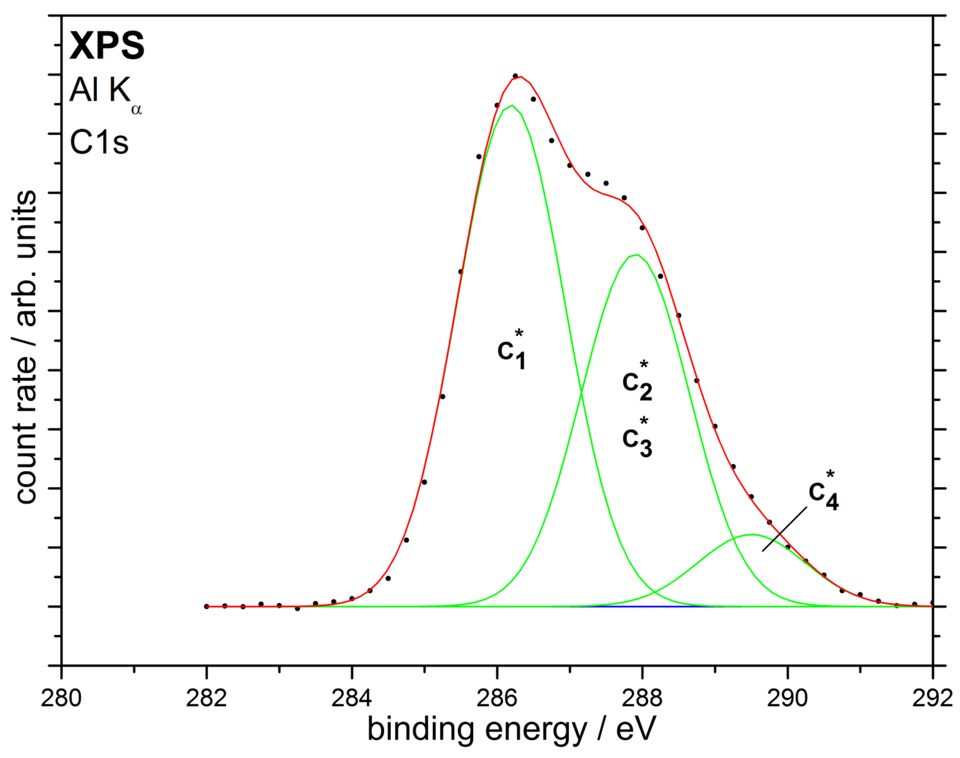
XPS 2nd step



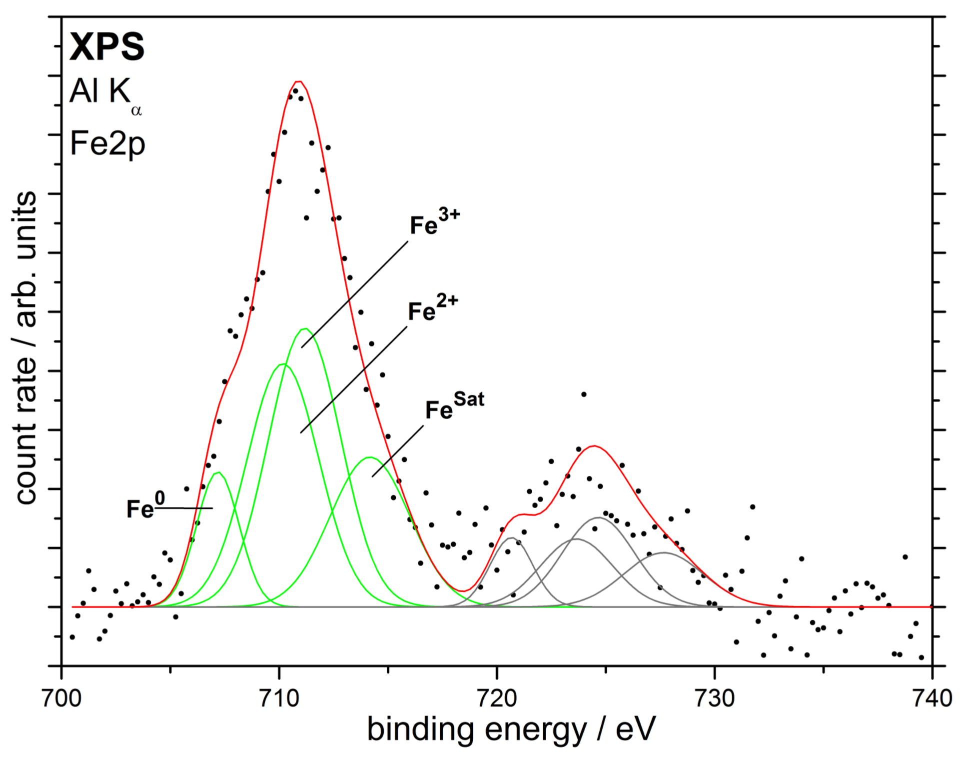
Orbital	Peak	rel. int.	Assignment
C1s	C_0	7.6 %	carbide
	C_1	50.4 %	C-C / C-H
	C_2	22.4 %	C-O
	C_4	19.6 %	C=O
Fe2p	I	15.3 %	Fe^0
	II	7.0 %	Fe^{2+}
	III	77.7 %	Fe^{3+}



XPS 3rd step



Orbital	Peak	rel. int.	Assignment
C1s	C_0^*	-	-
	C_1^*	54.2 %	C-C / C-H
	C_2^* & C_3^*	38.0 %	C-O & epoxy
	C_4^*	7.8 %	C=O
Fe2p	I	13.3 %	Fe^0
	II	40.4 %	Fe^{2+}
	III	46.3 %	Fe^{3+}



Conclusion

The present study explores insights into the adhesion mechanisms between stainless steel (316L) and industrial, polymeric epoxy adhesives. It is shown how surfaces change topographically as well as in their chemical composition, if epoxy and steel layers get into contact with each other:
Even very carefully and under ultrahigh vacuum conditions cleaned steel surfaces still exhibit significant traces of carbon and oxygen. The interaction between the two materials results in various functional carbon groups like C-C, C-O and C=O and in the reduction of iron at the steel surface from Fe3+ to Fe2+ according to the different preparation steps. This reduction occurs probably due to the interaction between the functional groups of the epoxy resin and iron oxides. Spin-coating the epoxy resin forms a layer on the steel surface including smooth regions as well as areas covered by clustered epoxy.

Literature

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Atom- und Molekülphysik an Oberflächen