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High resolution depths profiling using 194nm femtosecond laser ablation coupled to ICP-MS and applied to Sn coating analysis

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The analysis of thin layers especially of metal coatings are of wide interest for many industrial applications. Sn coatings are widely used in the food packaging industry for corrosion protection, and in the electronics industry in soldering applications. Food industrial standards have restrictions with respect to Pb concentrations as described by EU regulation No 466/2001. For example, Pb should not exceed 0.02 mg/kg in cow milk or 1.5 mg/kg in bivalve moluscs. Lead in food contact materials (FCM) mainly occurs in tin and pewter (tin based cutlery) and old drinking water pipes from the 1900s. So far there are only general requirements stated for FCM in EU regulation No 1935/2004 and a maximum concentration for Pb in FCM has not been defined.

Electrochemical plated test samples for food packaging with a measured tin coating of 22g/m^2 to 1.9g/m^2 related by the density of Sn with 7.31g/cm^3 to a thickness of 1.5µm - 0.26µm have been analyzed by UV femtosecond laser ablation coupled to ICP-MS. The primary goal was to determine the Pb concentrations as a function of depth. An ablation procedure allowing the removal of single layers over larger areas of any mathematical defined shape has been developed in order to remove layers down to a thickness of 30 nm over a time interval suitable for the precise determination of major and trace elemental concentrations. Data for 10 subsequent analyses (layers) representing the tin layer plated on a steel base have been analyzed using different energy densities of the applied laser pulse. The corresponding laser ablation craters have been measured for their depth using confocal laser scanning microscopy capable of a resolution of 10 nm. The depth profiles appear as binary mixing of iron and tin but in fact reflect the surface roughness of the steel base. In all measurements Pb-content are low throughout the profile at 0-5µg/g and strongly increase towards the surfaceof the plated layer reaching concentrations up to 85µg/g. Electrochemically, Pb is following the Sn due to its slight potential differences. The concentrations of Pb should therefore depend on the thickness plated onto the steel base.