Characterization of Aerosol Generated by Laser Ablation for Inductively Coupled Plasma Mass Spectroscopy.

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CHARACTERIZATION OF AEROSOL GENERATED BY LASER ABLATION FOR INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY

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INTRODUCTION

Laser ablation (LA), together with inductively coupled plasma mass spectrometry (ICP-MS) as a detection system, has become a routine method for the direct analysis of various solid samples. The product of laser ablation contains a mixture of vapour, droplets and solid particles. All components are finally transported to a plasma by a carrier gas in form of dry aerosol that includes mainly agglomerates of primary nanoparticles. Dry aerosol can be studied by different offline or online techniques. In general, characterisation of aerosols by their particle size distribution (PSD) represents indispensable tool for fundamental studies of the interaction of laser radiation with various materials. Many works have studied PSD of dry aerosol for different samples, and different ablation conditions (Hola *et al.*, 2010).

The laser ablation was performed with nanosecond laser (193 nm) on glass and steel samples using various ablation conditions (spot size, ablation mode, repetition rate, fluence). The aerosol characterization was made first by online PSD monitoring using Engine Exhaust Particle Sizer (EEPS) simultaneously with laser ablation - ICP-MS analysis. Second, the structure of the laser-generated particles was studied off-line using scanning electron microscopy (SEM) and atomic force microscopy (AFM).

EXPERIMENTAL SETUP

The particles produced by laser ablation of standard materials (glass NIST 610 and steel F4) were analysed by ICP-MS and various aerosol spectrometers (EEPS, APS and OPS) giving information about the physical properties of generated particulates. The arrangement of the experiment is shown in Figure 1.

The instrumentation of the LA-ICP-MS system consisted of an excimer laser ablation system Analyte G2 (Photo Machines Inc., Redmond, WA, USA) and ICP-MS with a quadrupole analyzer Agilent 7500ce and a collision-reaction cell (Agilent, Japan). The laser operates at a wavelength of 193 nm with a pulse duration ≤ 4 ns. Using helium as a carrier gas with a flow rate of 0.65 l min^{-1}, the aerosol was washed out from the chamber (HelEx) and transported through a polyurethane tube (i.d. 4 mm) to the aerosol

spectrometers and ICP-MS. Two ablation modes - spot and line scan - were performed. Spot ablation with different spot sizes and line scan ablation using 110 μm spot size and different scan speed were compared. Selected isotopes were monitored with the total integration time of 1 s which was similar to the EEPS scanning rate.

EEPS spectrometer is an aerosol instrument allowing to measure number size distribution in fixed particle size range of 5.6 – 560 nm with a high time resolution (down to 1 second per sample). The EEPS classifies the particles according to their mobility in electrostatic field and counts their number using set of 22 electrometers.

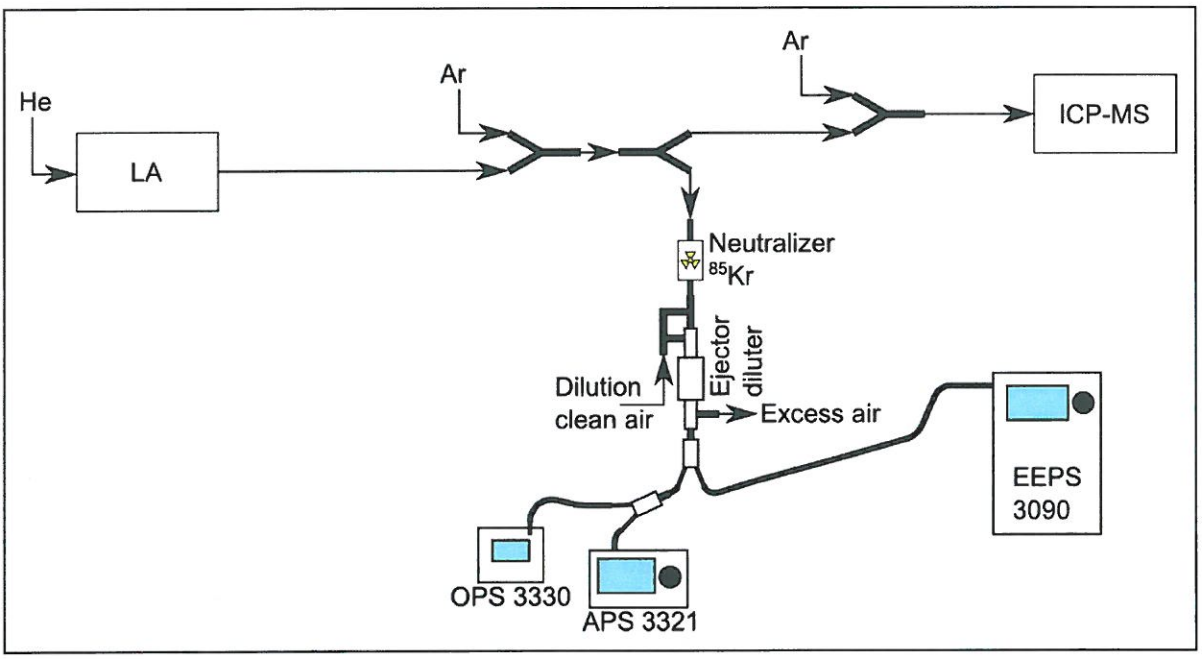

Fig. 1: Schematics of measurement set-up

The post-situ off-line measurements were made by SEM/AFM method after their collection on a polycarbonate membrane filter and Highly Oriented Pyrolytic Graphite (HOPG) substrate. The mesurements were performed first using Bruker Icon AFM with ScanAssyst-Air probe in PeakForce QNM mode. Later, simultaneous surface characterization by SEM and SPM in the same coordination system (LiteScope SPM, Nenovison) was performed. Collected data was evaluated using Gwyddion free SPM analytical software (Nečas and Klapetek, 2012).

RESULTS AND CONCLUSIONS

The average size distributions (measured by EEPS for 80 s laser ablation) show very similar multimodal shape for both materials and both ablation modes (see Fig. 2). Spot ablation mode of both materials produces higher concentration of primary particles and lower concentration of largest particles compared to line ablation mode. The smaller particles represent the primary particles produced by laser ablation, the larger particles are most probably a product of coagulation/agglomeration of primary nanoparticles or particles originated from the droplets' solidification (Novakova *et al.*, 2016).

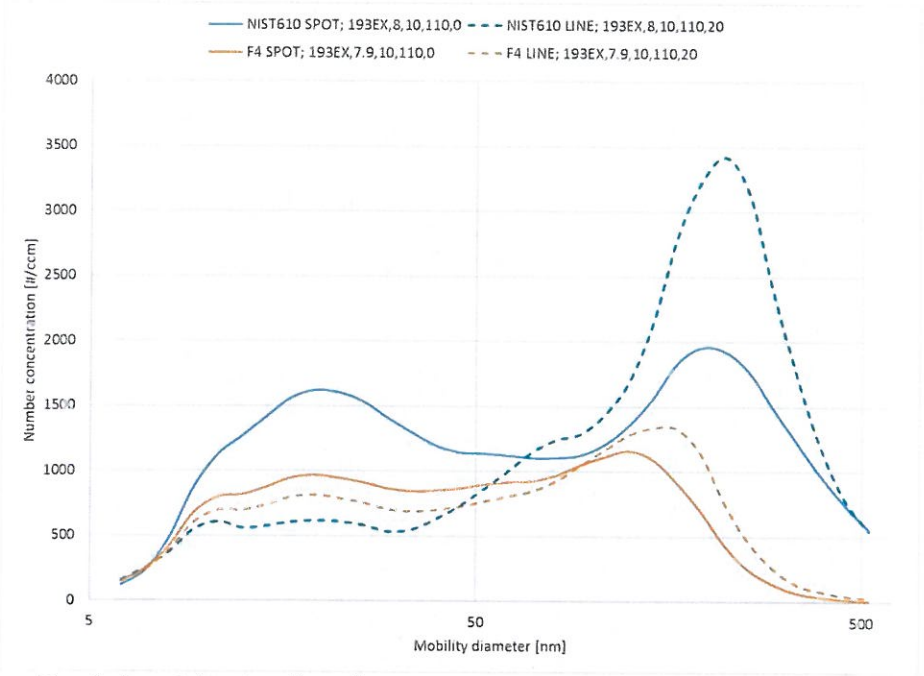

Fig. 2: Particle size distribution graphs (average of 80 s ablation).

The EEPS measurements can confirm different time resolved particle formation for different ablation modes. While the line ablation mode provides stable particle formation, the PSD is changing during ablation within one spot as shown in Fig. 3 for the NIST 610.

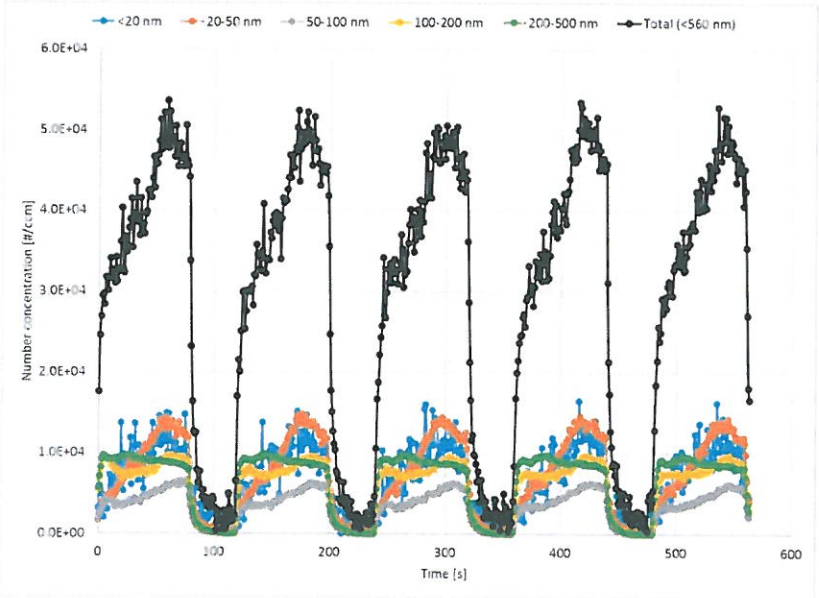

Fig. 3: Spot laser ablation (5 repetitions).

The structure of the agglomerated nanoparticles were visualised by SEM and AFM method. As an example of AFM neasurement, particles from the spot laser ablation of the steel sample are shown in Fig. 4.

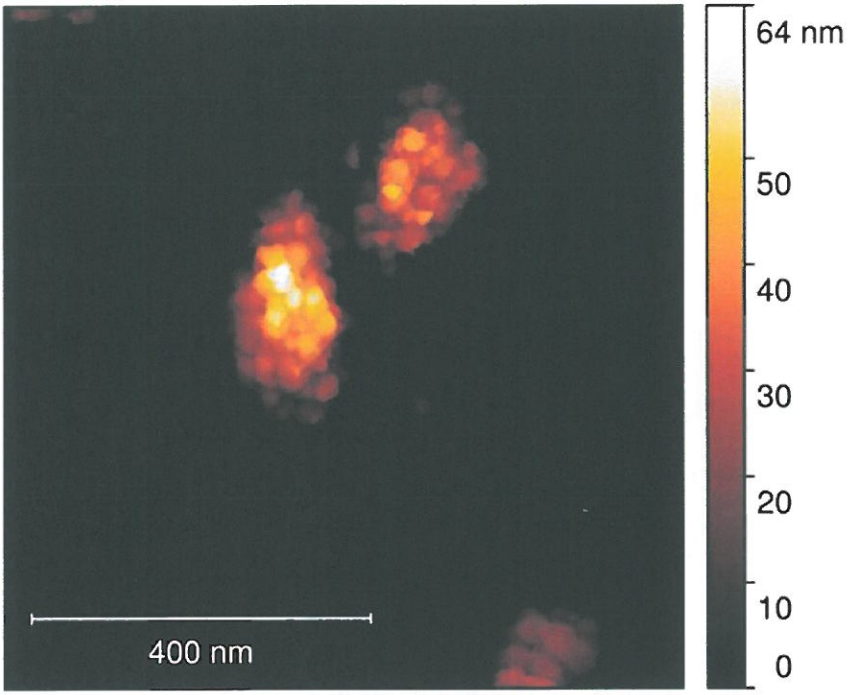

Fig. 4: AFM measurement of agglomerated nanoparticles.

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REFERENCES

Hola M., Konecna V., Mikuska P., Kaiser J., Kanicky V., Influence of physical properties and chemical composition of sample on formation of aerosol particles generated by nanosecond laser ablation at 213 nm, *Spectrochim. Acta B*, 65, 51–60, (2010).

Nečas D. and Klapetek P., Gwyddion: an opensource software for SPM data analysis, *Central European Journal of Physics*, 10, no. 1, 181-188, (2012).

Novakova H., Hola M., Vojtisek-Lom M., Ondracek J., Kanicky V., Online monitoring of nanoparticles formed during nanosecond laser ablation, *Spectrochimica Acta Part B*, 125, 52–60, (2016).