Properties of Aerosol, Produced by Laser Ablation of Standard Materials for ICP-MS Analysis.

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PROPERTIES OF AEROSOL PRODUCED BY LASER ABLATION OF STANDARD MATERIALS FOR ICP-MS ANALYSIS

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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 (Figure 1). All components are finally transported to a plasma by a carrier gas as a dry aerosol including mainly agglomerates of primary nanoparticles. 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.

The particle size distribution of dry aerosol originating from laser ablation of standard material was monitored by two aerosol spectrometers – Fast Mobility Particle Sizer (EEPS) and Scanning Mobility Particle Sizer (SMPS) simultaneously with laser ablation - ICP-MS analysis.

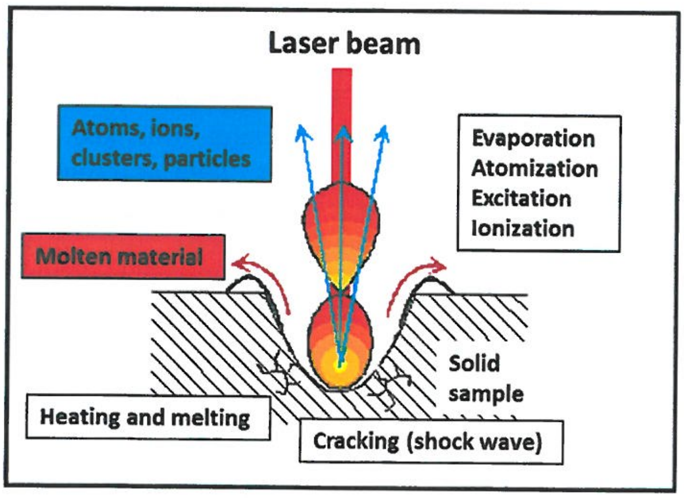

Fig. 1: Laser beam-sample interaction.

EXPERIMENTAL SETUP

The particles produced by laser ablation of standard materials were analysed by ICP-MS and various aerosol spectrometers giving information about the physical properties of generated particulates. The arrangement of the experiment shows Figure 2. This work is focused on the comparison of EEPS (model 3090, TSI) and SMPS (EC 3080, DMA 3081 and CPC 3775, all TSI) results.

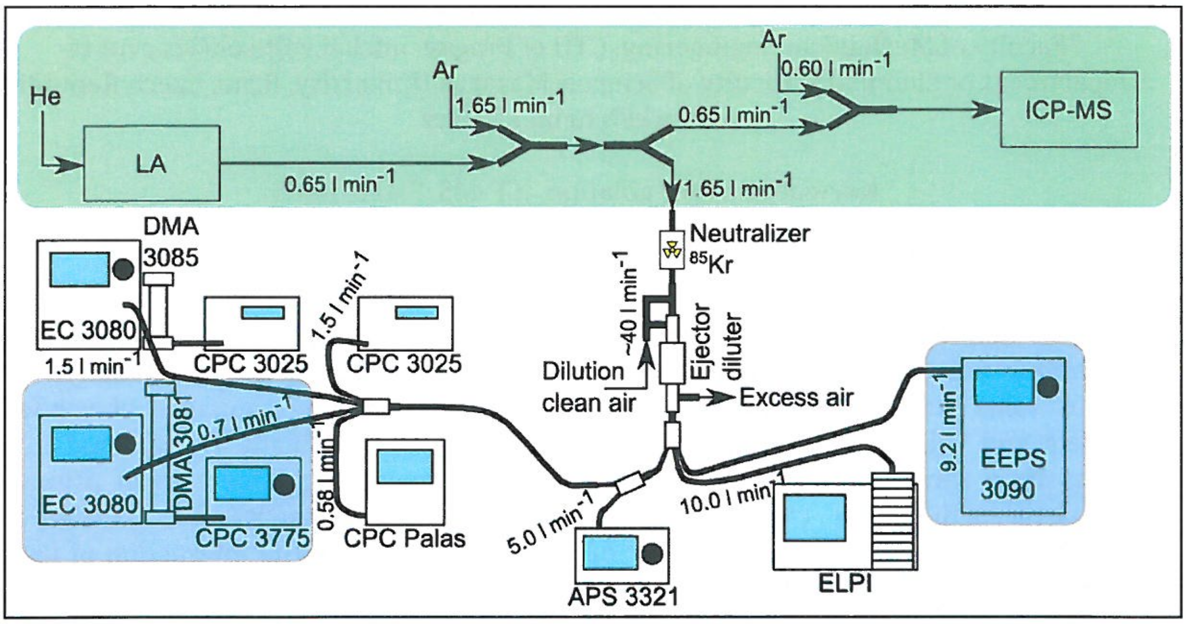

Fig. 2: Schematics of measurement set-up
(green – LA-ICP-MS system, blue – aerosol spectrometers used for this study).

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 85 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 FMPS scanning rate.

SMPS spectrometer is an aerosol instrument measuring particle number size distribution in size range starting at units of nanometers up to approximately 1 micron. This instrument sizes the particles according to their mobility in electrostatic field and counts their number in individual size bins using Condensation Particle Counter (CPC). SMPS 3936 including EC 3080, DMA 3081, aerosol charger ^{85}Kr (10 mCi) and CPC 3775 (all TSI) was used in this work. The measurement size range was set to 10 – 300 nm and the scanning time was set to 1 minute per sample.

EEPS spectrometer is another 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 again sizes the particles according to their mobility in electrostatic field and counts their number using set of 22 electrometers. The Engine Exhaust Particle Sizer (EEPS, Model 3090, TSI), which is, according to the manufacturer, functionally similar to the FMPS (Model 3091, TSI), was used for this work.

RESULTS AND CONCLUSIONS

The particle size distribution was monitored using SMPS and FMPS simultaneously with ICP-MS signal for the spot and line ablation. The records of PSD for the whole ablation time (80 s) were compared. Fig. 3 shows PSD for spot laser ablation, repetition rate 10 Hz, Fluence 8 J cm^{-2} and spot size of 85 μm. The error bars indicate standard deviation of 5 measurements.

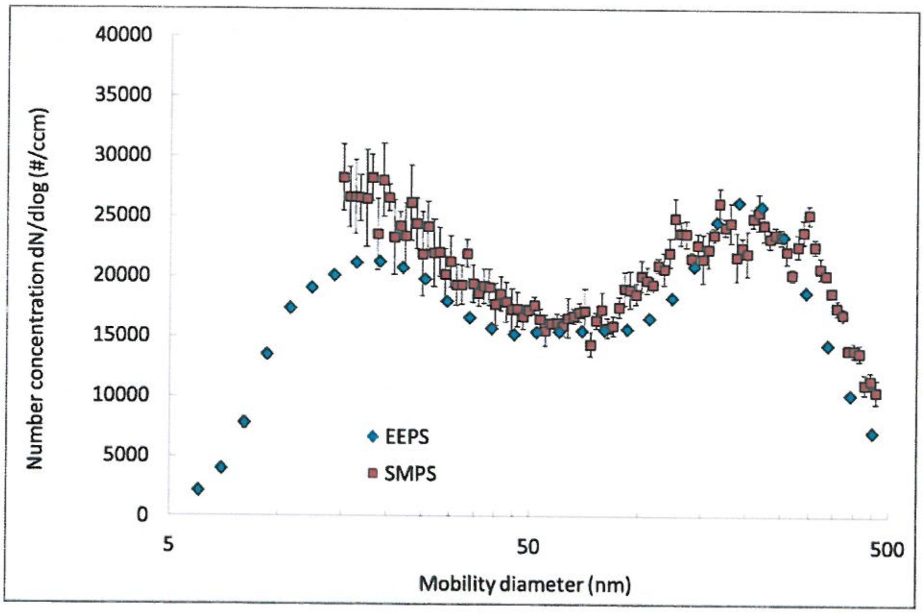

Fig. 3: Particle size distribution graphs (average of 80 s spot ablation).

Both size distributions are bimodal with the first mode in nucleation size range (10-22 nm) and the second mode in fine size range (190 nm). 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.

The FMPS records particle size distribution for each second of the entire ablation process using 32 size channels. To show some specific dynamic features of ablation and to demonstrate the high scan time of the FMPS device, maps representing the change in PSD over time were created. Figure 4 shows distribution maps for temporal behaviour of particle number concentration in individual size channels for spot size of 85 μm. The particle concentration scale is shown to the right dN/dlog(dp) (particles cm^{-3}).

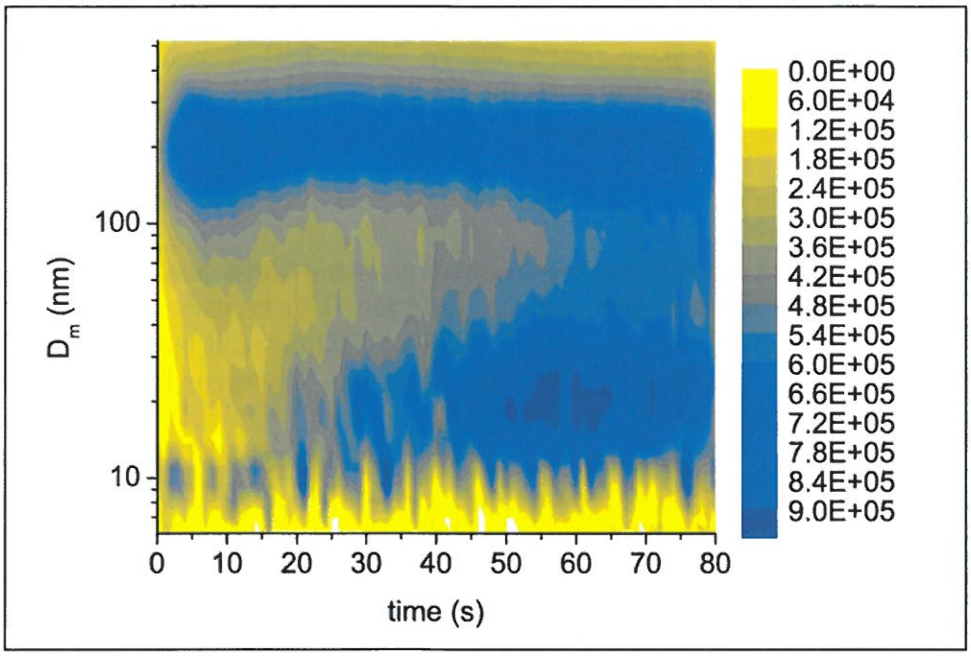

Fig. 4: EEPS distribution map.

Increased production of particles (Dm ~ 190 nm) was observed during single hole drilling within the first 10 s of laser ablation. The production of primary nanoparticles starts approximately 20 s after the start of ablation, which corresponds to a deeper crater. Furthermore, the number concentration of particles with Dm < 50 nm rises with increasing crater depth. Such behaviour at the beginning of the surface layer ablation confirms the theory of nanoparticles being scavenged by larger particles of the standard material. When the crater becomes deeper, the production of larger particles is diminished and the relative concentration of smaller nanoparticles thus increases. Generally, the spot ablation is dynamically changing during the ablation process.

ACKNOWLEDGEMENT

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