**Application of Laser-Induced Breakdown Spectroscopy**

**for the determination of trace metals in oils**

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The subject of this research is a method for preparation and analysis of oils by Laser-Induced Breakdown Spectroscopy (LIBS), aimed to minimize the necessary sample volume and the matrix effect while maximizing the detection sensitivity and measurement’s repeatability. The preparation procedure consists in stabilizing the oil sample and silica wafer substrate at a fixed temperature (40°C) and in delivering an oil droplet on the wafer rotated by a spin coater. In this way, an uniform oil film is obtained, which thickness is controlled through the rotational speed.

During the sample preparation, above a certain rotational speed the thickness of the oil film is the same for the two oils although their kinematic viscosities are very different, meaning that the volume sampled by LIBS is the same. The measured oil transmissivity at the laser wavelength of 1064 nm significantly decreases with concentration of impurities, but this effect could be neglected when dealing with very thin oil films. The plasma formation threshold measured on the bulk oil samples decreases with the impurity content. In case of pure oil, also for the maximum laser energy here used (165 mJ), the plasma is mainly initiated on the wafer, while the presence of impurities increases screening of the substrate by the plasma formed directly on the oil. The matrix effect disappears on a very thin film, here of 0.74 μm, where the C I line intensity in plasma does not vary with the total concentration of impurities; simultaneously, the plasma emission becomes stable from one laser pulse to another, contrary to the case of a thick liquid layer. In the optimized experimental conditions, the plasma emission from oil was very intense although the sample volume probed by each laser pulse was of 0.3 nL only. The obtained results show that the LIBS, with the proposed procedure of forming a thin layer of oil, can be very successfully applied for the quantitative analysis. Very low detection limits (in the range of 0.1 to 5 ppm) were obtained with good linearity of calibration curves in the range of 0 to 60 ppm for all tested elements [1].

Spatially and temporally integrated spectral measurements were performed under experimental conditions optimized for elemental analysis of trace metals in oil. Time-resolved values of the spectral line intensities, electron number density, and plasma temperature were obtained by subtracting averaged spectra recorded at different time delays. The electron number density was estimated using the Stark broadened profile of the Hα line. Ionization temperatures were derived from Mg ionic to atomic line intensity ratios. Spectra of C2 and CN molecules were used to evaluate the rotational and vibrational temperature of heavy particles. It is shown that the plasma parameters depend on the total metal concentration in the oil: although the effect is small, it must be taken into account in the quantitative determination of trace metals in the oil using the LIBS technique [2].

REFERENCES

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