Laser ablation plasmas for diagnostics of structured electronic and optical materials during or after laser processing

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ABSTRACT

Laser induced plasma can be used for rapid optical diagnostics of electronic, optical, electro-optical, electro-mechanical and other structures. Plasma monitoring and diagnostics can be realized during laser processing in real time by means of measuring optical emission that originates from the pulsed laser-material interaction. In post-process applications, e.g., quality assurance and quality control, surface raster scanning and depth profiling can be realized with high spatial resolution (~10 nm in depth and ~3 μm lateral). Commercial instruments based on laser induced breakdown spectrometry (LIBS) are available for these purposes. Since only a laser beam comes in direct contact with the sample, such diagnostics are sterile and non-disruptive, and can be performed at a distance, e.g. through a window. The technique enables rapid micro-localized chemical analysis without a need for sample preparation, dissolution or evacuation of samples, thus it is particularly beneficial in fabrication of thin films and structures, such as electronic, photovoltaic and electro-optical devices or circuits of devices. Spectrum acquisition from a single laser shot provides detection limits for metal traces of ~10 μg/g, which can be further improved by accumulating signal from multiple laser pulses. LIBS detection limit for Br in polyethylene is 90 μg/g using 50-shot spectral accumulation (halogen detection is a requirement for semiconductor package materials). Three to four orders of magnitude lower detection limits can be obtained with a femtosecond laser ablation – inductively coupled plasma mass spectrometer (LA-ICP-MS), which is also provided on commercial basis. Laser repetition rate is currently up to 20 Hz in LIBS instruments and up to 100 kHz in LA-ICP-MS.

Keywords: Laser ablation, material processing, optical diagnostics, chemical analysis, LIBS, LA-ICP-MS.

1. BASICS OF LASER ABLATION

Laser ablation is a simple technique for practical implementation; it offers rapid micro-analysis without a need for sample preparation. Any sample (solid or liquid) can be analyzed for elemental and isotopic composition using LIBS or LA-ICP-MS instruments. Additionally, molecular and structural characteristics of the sample can often be inferred using chemometric processing of the spectra. Built-in intrinsic features, such as a laser beam shaper/homogenizer and an autofocus function, enable high spatial (3-dimentional) resolution and uniform power distribution at the ablation spot that results in high precision measurements during rapid laser scans over the sample topography. These features significantly improve the reproducibility of the analysis. Time required for the sample loading, measurement and chemometric analysis of the acquired data is minimal – usually only a few seconds.

The major mechanisms of the formation and evolution of laser ablation plasma are well understood and can be predicted based on models. With femtosecond ablation pulses, the duration of the laser field is sufficient to heat up only the electrons within the ablated spot on the sample surface. An explosion-type escape of these electrons causes simultaneous destruction and ejection of the 'cold' lattice material due to the Coulomb forces. In the femtosecond mode (≤1 ps), the process of instantaneous vaporization and ionization of the sample can be simple, without inducing any heat transfer across the sample and any shielding of the laser irradiation by the plasma plume. The plume forms only after the end of the femtosecond laser pulse. In practice, significantly less expensive nanosecond lasers are often used, particularly in LIBS instruments. The nanosecond ablation frequently involves other entangled mechanisms that can be complex for scientific elucidation but remain applicable to viable chemical analysis. Matrix-dependent effects are often observed in nanosecond ablation. Depending on the objectives, we can either correct these matrix effects applying computational chemometric algorithms or exploit them for characterization of the matrices.

Short-wavelength (ultraviolet) lasers usually provide more efficient and reproducible ablation, and hence higher accuracy of the analysis relative to that attainable with infrared lasers. Advantages of the ultraviolet irradiation are

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associated with more efficient absorption in the sample bulk, smaller losses due to reflection from the surface and less absorption in the plume above the sample surface as compared to the laser-induced breakdown in the infrared. Because of the higher photon energy, the ultraviolet radiation can break lattice bonds even in a single-quantum absorption event.

Thus, short-pulse UV lasers generally provide the best performance metrics in terms of precision, accuracy, and sensitivity for chemical analysis, especially when ablation is coupled with the ICP-MS detection. In the latter case, the effects of elemental and isotopic fractionation (deviation in stoichiometry of the plume from that of the sample) are mitigated most efficiently further facilitating calibration, reproducibility and accuracy of the analysis. More details about LIBS and laser ablation can be found elsewhere.[1,2]

2. DIAGNOSTICS DURING LASER PROCESSING

Microelectronic, electro-optical and electro-mechanical devices are made of layered thin film structures with interconnected functional layers. Manufacturing of such devices often involve multiple steps of laser drilling, cutting, scribing, and patterning of the layers of different materials. Laser scribing is becoming a dominant technique for thin-film solar cell fabrication. Fabrication of micro-electro-mechanical systems (MEMS) demands comparable processing of various layers and structures made of foils, semiconductors, ceramics, plastic masks, and metal stencils. However, a laser beam can inadvertently damage also the underlying layer. Sometimes it is difficult to identify at which point a laser action must be discontinued to ensure a complete removal of the desired layer(s) but at the same time prevent any damage to the underlying structure.

Use of laser processing, in particular laser drilling and scribing with fast pulsed lasers, is expanding in the industry as it facilitates higher quality and faster production of electronic and similar devices. Lasers are ideal for processing brittle materials utilized in the solar and microelectronic industry. Lasers can be used to drill holes in silicon substrates to move the contacts to the back of the solar cell to avoid the 'shading' caused by the front electrodes. Edge isolation, soldering, semiconductor doping, and structuring active layers (i.e. scribing) are common applications where lasers are being used.

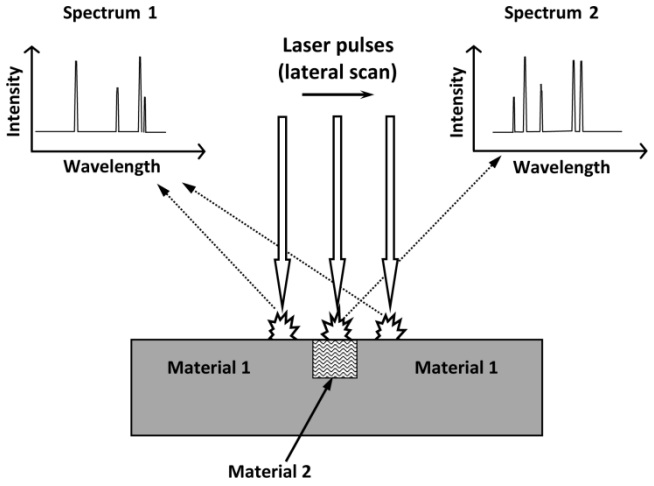

Fig. 1. Diagram of the laser ablation processing across a structure that comprises schematically of two materials.

Optical emission measurements are well suited for in-situ metrology and diagnostics during pulsed laser processing of materials in real time. Optical emission provides excellent specificity to chemical composition of the processed material and can be measured very fast. Optical measurements do not require vacuum and can be implemented contactless from a stand-off distance. A technique of optical diagnostics can be fully automated to enable high efficiency and throughput in manufacturing at best attainable precision and minimization of damage to the products. This technique includes measuring the optical spectral emission from the laser-ejected material and then using this obtained information to identify at least partially chemical composition of the processed material. A change in chemical composition indicates that laser ablation has reached a different layer or domain of the structured material. Such diagnostics can be used in situ to monitor specific chemical information during material processing to control or examine desired depth or lateral length of the laser processing.

Fig. 1 illustrates how a focused laser beam is directed at and scanned across a processed surface to generate consecutive pulsed ablation plumes of excited ejected material. Optical emission from these plumes is collected by an optical system, e.g. a lens and a fiberoptic cable, then recorded by an optical spectrograph typically fitted with a CCD or ICCD camera. The obtained spectral information is digitized and forwarded to a system computer for computational chemometric treatment of data to characterize and discriminate in real time between the two different constituents of the

structured material shown in this example. Recorded optical emission spectra from each consecutive pulse are characteristic of the ablated material at a corresponding location. The spectrum provides a signature of the chemical species present in the sample and their relative abundances. Optical diagnostics can be performed from a stand-off distance without direct contact with the ablated material. Therefore, no disturbance or interference with the material processing is required to implement this optical diagnostic method. The stand-off distance is usually small to facilitate efficient collection of the optical emission.

Although Fig. 1 shows laser pulses directed in different lateral locations on the surface of the structured material, it is also possible to direct the consecutive laser pulses in the same location, thus ejecting the ablated material from consecutive depth points, with each depth point being successively deeper. In this fashion, the layer-by-layer analysis can be performed. The spectra presented in Fig. 2 demonstrate how optical emission from successive laser ablation plumes during laser drilling can be used to obtain chemical information and identify consecutive layers of the film stack structure of a solar cell based on copper indium gallium diselenide (CIGS). Each successive laser pulse was fired into the same spot, forming a crater deeper and deeper in the stack structure. The recorded series of spectra corresponds to the consecutive layers of the stack. The CIGS photovoltaic structure used in this example had a stack of CIGS, Mo, Ti, and Al layers, from top to bottom. The displayed series of spectra corresponds to the consecutive layers of the structure. The recorded optical spectra indicate presence of Al, Ga, In, Mo, and Ti in different layers. Using this information, their chemical composition and homogeneity, and the thickness of the layers can be determined and monitored in real time.

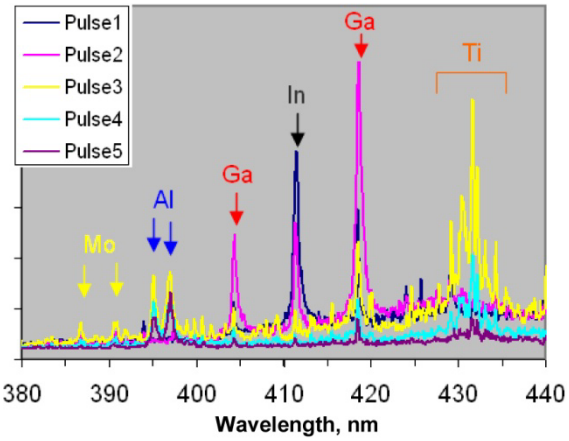

Fig. 2. Optical emission spectra from successive laser ablation plumes during laser drilling of a CIGS-based solar cell structure.

The CCD or ICCD camera can be synchronized with the individual laser ablation pulses in such a way that optical emission is measured with an appropriate delay after a laser pulse and only during a gated period of time. The delay and the gate width can be optimized to enhance useful spectral features while concurrently decreasing continuum background in laser-generated optical spectra. When a pulsed laser with high repetition rate is used for material ablation, the fast on-chip accumulation of spectral data in the pixels of the CCD or ICCD camera integrated over multiple laser pulses can be used to increase signal-to-noise ratio of the recorded spectra, while enabling a fast gate capability. On-chip accumulation refers to adding up multiple exposures to integrate signals from many photons in the pixels on a CCD detector chip before they are read out. It is possible to transfer the integrated charge quickly under the CCD storage areas. Alternatively, spectral data from each individual pulse can be measured to detect a change of chemical composition of the material in a technique often referred to as "end-point detection."

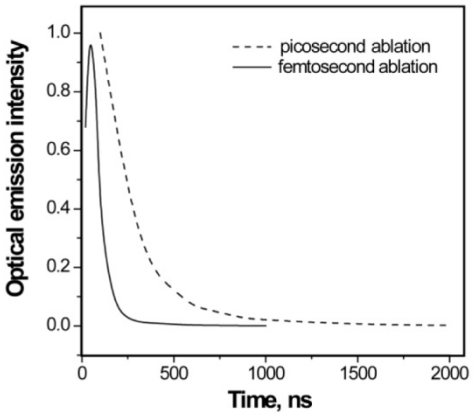

Fig. 3. Lifetime of the optical emission generated by ablation with picosecond and femtosecond lasers.

In industrial processing of materials, pulsed lasers with high repetition rates are commonly utilized. Typically, energy output of such lasers is limited to microjoule or low millijoule levels per pulse. Otherwise the high-repetition nanosecond lasers are used for drilling of vias and scribing of solar cells and semiconductor processing; such lasers also have typical output energy of ~1 mJ or less. As an example, the evolution of the optical emission generated by ablation of copper with picosecond and femtosecond lasers is depicted in Fig. 3. The lifetime of the optical emission is shorter than 1 μs. Therefore, the optimal delay of gated data acquisition should be less than 1 μs during typical industrial processing of materials with picosecond or femtosecond lasers. This is in contrast to relatively long-lived optical emission (several microseconds up to ~1 ms) that can be generated by ablation with low repetition rate lasers, which yield high energy pulses on the order of 10 mJ per pulse or higher.

Many integrated circuits are encapsulated in plastic packages that have external contacts to interface with the circuit. Similar plastic coatings are used on printed circuit boards. In traditional failure analysis of integrated circuits (to understand the failure mechanism and to determine whether a design change is warranted), the plastic is removed using a wet etch (acid) while avoiding further damage to the circuit. Unfortunately, this technique causes a significant amount of acidic waste and is excessively time consuming. Laser ablation can remove substantially any material on which a laser pulse impinges. A laser beam can be rapidly scanned in a two dimensional pattern across a surface to ablate the entire surface, for example, a plastic integrated circuit package. During this process, conventional laser stripping instrumentation cannot distinguish between the plastic and the underlying integrated circuit. Conventional laser stripping of the plastic package or coating may either result in not exposing the integrated circuit or damaging the circuit, neither such result provides the desired outcome.

By combining a scanable processing laser with an optical emission detection system and a control system, the laser can drill blind holes or be scanned across the multilayered electronic structures to remove the desired layers without inflicting damage to the underlying layers. The optical detection and analysis system can distinguish in real time between the material to be removed from the underlying materials. As soon as the material from the underlying layer is detected in the laser ablation plume by the optical diagnostics system, the laser is stopped.

Chemometric analysis of digital spectral data can be implemented to identify and characterize the ablated material on the basis of the measured spectra. The chemometric algorithms can compare a real-time measured spectrum to the spectra stored in a reference database in memory of a system computer. This method allows us to identify, discriminate and characterize the ablated materials in a real-time automated mode of operation. Materials of similar elemental composition but of different molecular structure can be discriminated with this method. For example, we applied a combination of principal component analysis (PCA) and partial least squares (PLS) algorithms to identify a variety of complex organic and inorganic materials.[3] Some physical properties of the materials, such as reflectance or density, and certain crystallographic structures can also be discriminated in many cases. During the process, material compositional and structural consistency or deviations from the standard sample can be monitored in real time. Traditional one-element calibration or multivariate chemometric procedures can be applied for elemental quantification.

Functional material structures are often three-dimensional. For example, the upper surfaces of microelectronic devices, integrated circuits, photovoltaic cells, micromechanical systems are all not planar. Moreover, electrical contacts such as bond wires or flip chip solder bumps are typically raised above the surface of the electronic devices. The control system computer can include a memory and a raster locator. The laser can be scanned over the entire surface of the electronic device until a first location is detected and stored for which material of the layer of interest is detected. On a next scan the laser can be togged off for that location. In a removal of an overlaid plastic packaging cover, for example, the scanning of the laser can continue until an entire upper surface of the device is exposed, yet remains undamaged by the removal of the upper layer.

3. POST-PROCESS ANALYSIS

An ability to perform rapid microscopic chemical mapping and depth profiling by laser ablation readily lends this technique to multiple applications in the areas of quality assessment and quality control, as well as for identification and characterization of possible defects. During analysis, spectral data are promptly analyzed using statistical chemometric algorithms that facilitate chemical identification of elemental and some of the structural composition. LIBS precision is usually 2-4%, while LA-ICP-MS precision can be <1%. Both techniques are usually considered as elemental analytical methods. However, using the matrix effect as a benefit, LIBS can discriminate compositionally similar organic compounds (we demonstrated earlier the capabilities to characterize various motor oils, fuels, lubricants, thermoplastic polymers, drugs, inks, explosives and propellants).[3] Similar analysis can be applied to detection of organic solvents, photoresist and polymers that are widely used in the semiconductor industry. The variations in LIBS spectra for different materials with similar elemental compositions are due to differences in the materials' light absorption properties and the optical power required to break their molecular bonds. These and other mechanisms determine the physical state of the laser-induced plasma and therefore, the resulting spectra.

Commercial laser ablation instruments enable rapid analysis of final manufactured products (or their parts) off-line but still in real time. As an example of using LIBS system RT100 (Applied Spectra) for depth profile monitoring during

fabrication of the metallic thin films in solar cell structures made by roll-to-roll printing of nanoparticles, we measured the relative intensities of spectral lines of Mo, Ti, and Al depending on the ablation crater depth. In this case, several laser pulses were directed to the same location, at which a deeper and deeper crater was progressively formed, thus performing the layer-by-layer analysis. The results are plotted against the consecutive laser pulse number in Fig. 4. The energy of nanosecond laser pulses (operating at wavelength of 532 nm) was adjusted so that the first 7 pulses removed the top CIGS layer, while the other 7 pulses were used to analyze metallic films. These settings were optimized to provide the necessary depth resolution at the highest speed of analysis. Figures 4(a,b) represent a high-quality product, in which the metallic thin films are printed on a substrate with the specified thickness of 200 nm. Each point in Fig. 4(a) is an average of four lateral locations on the sample with a relative standard deviation below ±0.04. Similar data are presented in Fig. 4(b) but for a single location, without averaging. Fig. 4(c) illustrates a defected location where the first metal layer is too thin and the second metal layer appears too close to the surface of the photovoltaic structure. Only 14 laser pulses were used for each profile in Figures 4(b,c) while providing sufficient information on the quality of the product and nature of the defects. Finally, the samples were scanned across, performing the depth profile at every location within approximately 1 sec.

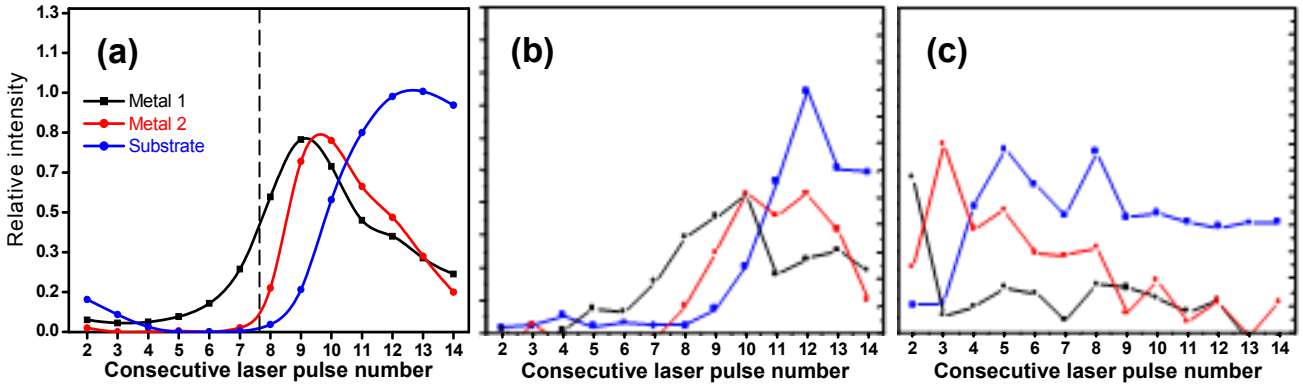

Fig. 4. Depth profiling analysis of solar cell films using commercial LIBS; a high-quality metallic thin film structure averaged over 4 locations (***a***); a high-quality location without averaging (***b***); and a defected film location (***c***). A vertical dashed line indicates an approximate position where the top CIGS layer ends.

Depth profiling was also performed on multilayered film stacks of the blue InGaN/GaN light emitting diodes (LED). The luminescence efficiency of these devices is particularly sensitive to the Mg doping profile. The structure includes a InGaN/GaN multiple quantum well layer, a thin AlGaN:Mg electron-blocking barrier, and a highly Mg-doped GaN contact layer on top. These materials are transparent in the near infrared. Therefore, we used an ultraviolet 266-nm laser to ablate and analyze the LED samples. The ablation spot size was 130 μm in diameter, the depth resolution was 70 nm per pulse. The results of the depth profile analysis are shown in Fig. 5 as optical emission intensities at three atomic lines of Mg (279 nm), Al (396 nm), and In (410 nm) averaged over 25 locations on the sample. These data demonstrate sufficient layer-by-layer resolution of the LIBS measurements. Such measurements can be accomplished using a commercial LIBS system on the factory premises in real time. Thus, any deviation from the optimum fabrication process can be promptly detected and corrected. This is in contrast to a conventional practice of sending samples elsewhere to be analyzed with a secondary ion mass spectrometer (SIMS) that takes about 3 to 4 days of turnaround time.

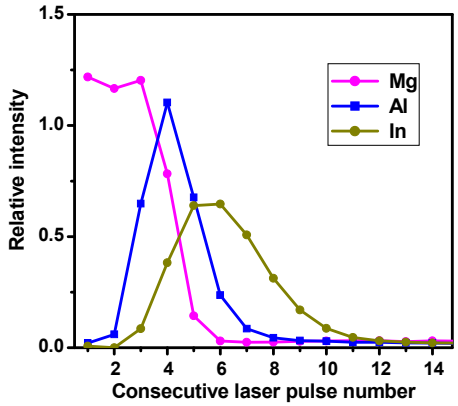

Fig. 5. Depth profiling analysis of the InGaN/GaN multiple quantum well LED structure using commercial LIBS.

Rapid chemical mapping by a LIBS instrument RT100 (Applied Spectra) was used to measure the sodium content distribution in photovoltaic CIGS films. Sodium doping is critical in improving CIGS solar cell efficiency, particularly to decrease resistivity by increasing carrier concentration up to an order of magnitude. However, the cumulative effect of Na on solar cell efficiency is process dependent and includes

multiple mechanisms that are influenced by parameters yet unidentified. Basically, the solar cell efficiency is enhanced for medium amounts of incorporated sodium, but decreased again for high Na doses. The adhesion of the CIGS film to the underlying molybdenum layer is also degraded when Na content is excessive. Consequently, monitoring the concentration and uniformity of sodium in CIGS films during the fabrication process is essential. We used a square grid of 50 ablation spots (70 μm in diameter, spaced by 150 μm) for rasterized scanning of the square CIGS samples of 2 × 2 mm in size. The intensity of Na emission at 589 nm was normalized by the intensity of the In line at 451 nm in order to improve precision of measurements and correct for possible laser ablation pulse-to-pulse irreproducibility. Three laser shots were applied at each location and the data were averaged to provide the relative standard deviation of ±0.05.

Generally, the use of laser beams for ablation has a fundamental limitation on the lateral resolution that is imposed by the diffraction limit. Light cannot be focused to dimensions smaller than roughly half its wavelength, which for conventional lasers is on the order of a few hundred to one thousand nanometers. It is possible to overcome this restriction using optical near-field effects. When a sample is placed in the near field, the laser radiation can be confined within evanescent or non-propagating fields. This approach results in sub-diffraction resolution that can become a basis for laser processing and analysis of materials on the nanoscale level. However, we have not yet obtained analytically useful optical emission in the near-field experiments. Using far-field optics with high numerical aperture for femtosecond ablation (100 fs pulses at 400 nm), we measured Na and K atomic emission within an ablation spot diameter of only 450 nm on a mica (muscovite) sample.[4] The emission lifetime was 20 ns for sodium at 589 nm and 30 ns for potassium at 766 nm.

Isotopic analysis is typically performed by mass spectrometry, including LA-ICP-MS. We developed a new approach to rapid isotopic analysis in ambient atmospheric air using LIBS instrumentation. This technique, Laser Ablation Molecular Isotopic Spectrometry (LAMIS), exploits ablating the sample and analyzing optical spectra from molecular species that are produced during plasma expansion into the air.[5] LAMIS broadens the capabilities of LIBS by adding optical isotope measurements from molecular spectra, which exhibit significantly larger isotopic shifts than atomic spectra. We measured spectra of diatomic radicals SrO, BO, OH, CN and C_2 in laser ablation plasma to demonstrate excellent spectral resolution and quantitative determination for several isotopes of Sr, B, C and H. For practical applications, LAMIS is poised to speed up, to simplify and to make isotopic analysis more affordable than now, while it will remain generally less sensitive than the traditional mass spectrometry.

Diverse samples can be analyzed for elemental and isotopic composition using LIBS or LA-ICP-MS. For LIBS, a multitude of applications in quality control during manufacture of electronic devices continues to expand including areas where LIBS is already proven to be beneficial (depth-resolved detection of phosphorus in electronic hard drive substrates; chemical identification of microdefects in silicon wafers, etc.). Many traditionally used metrology techniques are not practical for real-time diagnostic measurements in processing of the micro-scale structured materials. Other real-world applications for laser ablation include chemical analysis in ecology, geology and mining industry. Important applications are the detection of lead in children toys, paint coatings, electrical solders and other consumer products. Various pen and ink on paper can be identified for forensic applications.

Bulk analysis usually requires a relatively large amount of sample that must be sufficient to represent the mean elemental composition. Femtosecond ablation with a laser operating at high repetition rates enables fast scanning over a relatively large sample area and a high material removal rate. Direct sampling by laser ablation has many advantages over traditional solution nebulization into ICP-MS, including a smaller required sample size (~1 μg), real-time analysis, no acidic digestion, no contamination, no consumables and no waste. Accuracy and precision are similar to that obtained with nebulization because the nearly monodisperse nanosized aerosol particles from femtosecond ablation are readily atomized in the ICP without visible elemental or isotopic fractionation. At a repetition rate of 100 kHz, the area of 1 mm^2 can be scanned within 1.5 sec. During such scans, the mean ICP-MS signal at a particular element represents its average concentration in the sample, while variations of the signal reflect elemental distribution within the sample (homo- or heterogeneous). The latter information about sample homogeneity can be used for real-time quality control and quality assurance.

For illustration Figures 6 and 7 show the LA-ICP-MS data obtained from the glass and granite samples scanned using the 400-fs laser ablation system J100-UV (Applied Spectra). The data in Fig. 6 represent responses for ^{27}Al, ^{232}Th and ^{238}U from the perfectly homogeneous glass sample (NIST-612) collected using a 200-ms integration time of the

detector. The shaded intervals correspond to the 30-second periods when the ablation laser was running at wavelength of 343 nm and a pulse repetition rate of 20 kHz. The scan speed across the sample surface was 20 mm/s with laser-beam spot diameter of 6 μm, yielding the mass ablation rate of 0.4 mg/min. Wash-out time between these periods was used to purge the LA-ICP-MS system with a blank flow of gas (laser off). The intensities in Figures 6 and 7 represent the six areas on the sample (2 × 2 mm each) scanned in a rectilinear pattern with the total tested area of 24 mm^2 for each sample. The described analysis takes less than 7 min that includes measuring the blank before ablation starts.

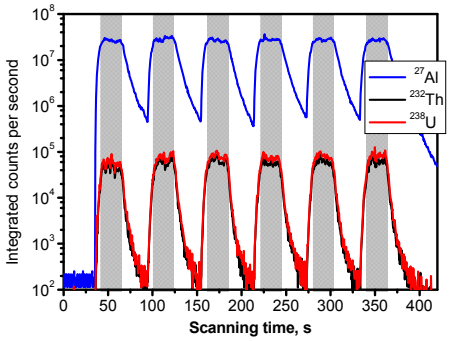

Fig. 6. High repetition rate femtosecond LA-ICP-MS data from the NIST-612 glass sample.

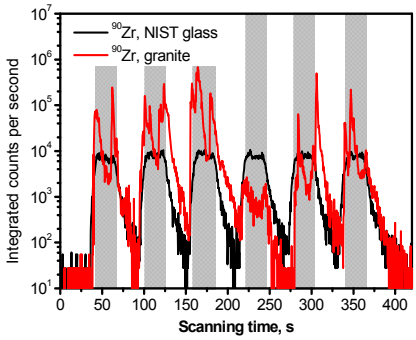

Fig. 7. Same as in Fig. 6, comparing Zr signals from the NIST-612 glass and the granite samples.

The NIST-612 glass sample exhibits a homogeneous elemental distribution including trace elements (concentration of ^{232}Th and ^{238}U was 37 μg/g). For a comparison, the responses of ^{90}Zr from both the NIST-612 and granite samples are shown in Fig. 7. The results of ablating granite portray a very inhomogeneous distribution of Zr, which is contributed mainly by the grains of minor mineral zircon ($ZrSiO_4$). Fig. 7 demonstrates how the homogeneous and inhomogeneous samples can be promptly differentiated for the purpose of quality control in industry. For the purpose of bulk analysis, the total ablation time and mass can be increased to better represent the bulk content in inhomogeneous samples as the larger ablation mass will improve both precision and accuracy of the analysis. The total amount of ablated mass from the granite sample was 1.2 mg, which falls short of representing the mean Zr content of this sample (nebulization into ICP-MS often requires at least 200 mg of grinded and dissolved granite to achieve a statically representative sample).

In summary, LIBS and LA-ICP-MS techniques are the most suitable for surface chemical mapping and depth profiling with high spatial resolution. They are universal, with no fundamental restrictions on the type of samples to be analyzed. LIBS can be used as a real-time process diagnostic method. Aerosol from high repetition rate ablation streamed into ICP-MS can provide performance metrics similar to liquid nebulization but without dissolution of samples. Our femtosecond laser ablation system is a bench-top instrument, while LIBS systems are available in briefcase and bench-top configurations with echelle or multi-channel spectrographs.[6]

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