

# Imaging of laser-induced microexplosions in dielectrics using time-resolved X-ray diffraction technique

## [1] Organization

Leader : Dr. Arturas Vailionis  
(Stanford Nano Shared Facilities, Geballe  
Laboratory for Advanced Materials)

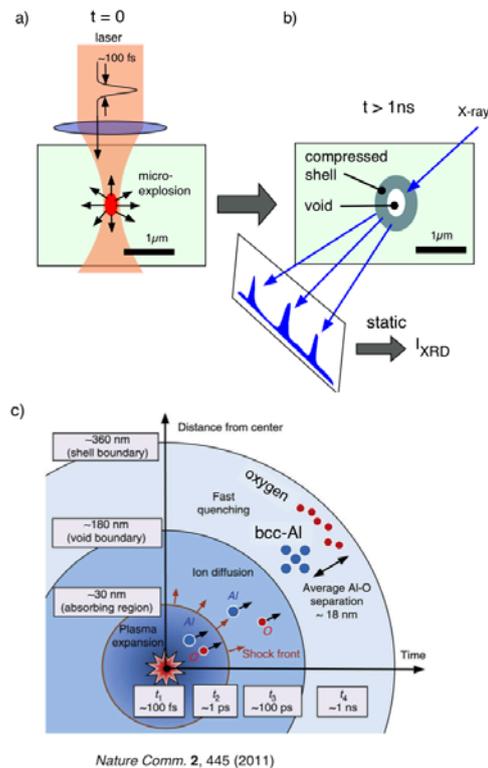
Representative : Prof. Vyngantas Mizeikis  
(Research Institute of Electronics, Shizuoka  
University, Japan)

Participants :

Prof. Akira Saito  
(Division of Precision Sci. & Technology and  
Applied Physics, Graduate School of  
Engineering, OSAKA University, Japan)  
Prof. Atsushi Ono  
(Research Institute of Electronics, Shizuoka  
University, Japan)  
Prof. Saulius Juodkazis (Centre of Microphotonics,  
Swinburne University of Technology, Melbourne,  
Australia)

## [2] Research progress

The purpose of this project is to use time-resolved monitoring of formation of high-pressure high-temperature (HPHT) phases of materials during their excitation by powerful ultrashort laser pulses. The principle of HPHT laser modification and main results achieved in sapphire ( $\text{Al}_2\text{O}_3$ ) are described in an earlier publication co-authored by some of the participants of this project (Vailionis, A., et al., "Evidence of superdense aluminium synthesized by ultrafast microexplosion", *Nature Communications* **2**, 445 (2011)). In sapphire, nanocrystals of super-dense aluminum phase were synthesized via extreme pressure and temperature conditions during laser-induced microexplosions. The new bcc-Al phase was identified from steady-state X-ray diffraction (XRD) characterization of laser-processed regions in sapphire. It was suggested that aluminum formation is due to spatial separation between oxygen and aluminum ions in a highly-excited plasma, which occurs



**Fig. 1.** The principle of LIMT (a), formation of a void surrounded by a shell of densified material, and detection of new phase using XRD (b), model of bcc-Al formation in compressed sapphire in temporal and spatial domains (c).

within the time scale of a few tens of picoseconds after excitation by a femtosecond laser pulse. Schematic explanation of bcc-Al laser formation during microexplosions is given in Fig. 1.

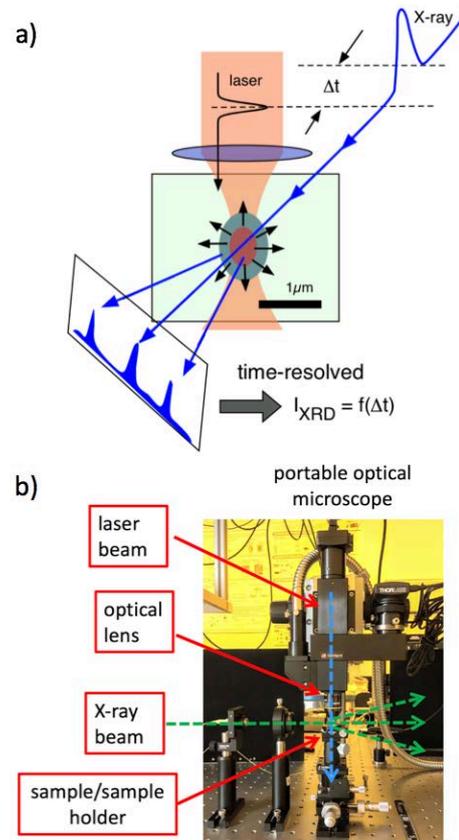
This research project is aimed at adding time resolution to the above XRD experiments, i.e., the ability to monitor transient XRD patterns by X-ray pulse passing the laser-affected region with a desired time delay after the laser pulse that initiated the microexplosion. By repeating microexplosion – pulsed XRD observation cycle at various delay times, temporal development of bcc-Al phase in sapphire can be visualized. More specifically,

goals pursued by this project are:

- 1) Development of novel characterization technique for time-dependent studies of Warm Dense Matter using ultrafast laser-pump x-ray-probe;
- 2) Understanding the creation and evolution of novel phases of matter under extreme conditions;
- 3) Development of knowledge and understanding of the effects of extreme conditions on materials performance and stability;
- 4) Practical use of laser-induced micro-explosion to create HPHT phases in materials other than sapphire.

### [3] Results

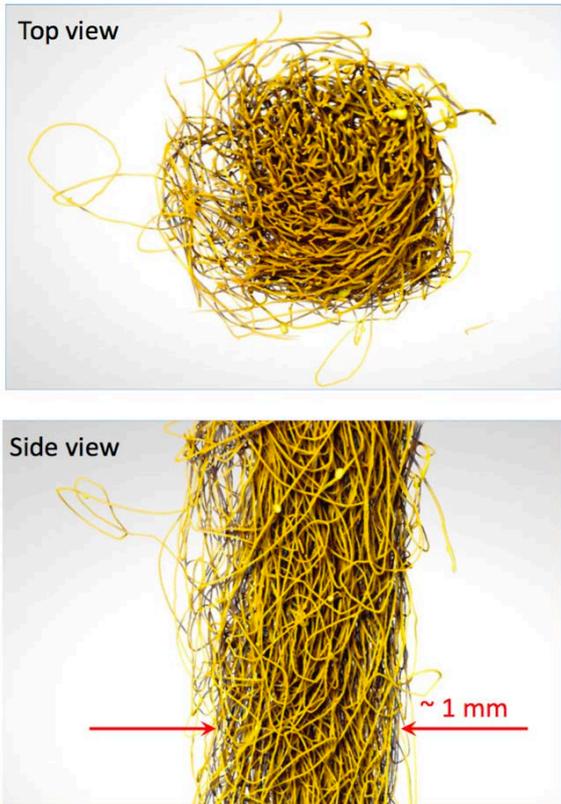
1) Opto-mechanical setup for time-resolved XRD experiments was developed. Its conceptual features are shown schematically in Fig. 2(a). The main feature of this setup is capability to overlap spatially a femtosecond laser pulse and an X-ray pulse in the sample under investigation, with temporal delay between the two pulses controlled by an optical delay line within the range from zero to a few nanoseconds. Each laser pulse exposes a new pristine region in the sample under investigation, while delay of X-ray pulse is varied systematically from negative (X-ray before the laser pulse) to positive (X-ray after the laser pulse). XRD pattern is recorded versus the delay, and it is expected that temporal development of X-ray diffraction pattern can be observed. From this development, formation of bcc-Al or other phases of matter under HPHT conditions can be visualized experimentally. The proposed experiments depend on the availability of temporally-synchronized fs laser and X-ray pulses, which is only available in a few facilities worldwide, one of them being Stanford University SLAC Linac Coherent Light Source. It is available on a competitive basis, i.e., scientifically sound application for X-ray beamline must be submitted, and approved for implementation by SLAC management body. Therefore, significant part of efforts during this year was directed towards the development of beamline application. If approved, Stanford University SLAC Linac Coherent Light Source facility will provide synchronized X-ray and optical pulses, whereas precise implementation of the interaction between the X-ray and optical pulse must be customized by the end user. Hence, it is necessary to prepare opto-mechanical setup that enables focusing of the fs laser beam into the sample investigated. The setup must be portable, i.e., can be carried to the X-ray facility. For this purpose, a compact



**Fig. 2.** Fig. 2 Dynamic monitoring of modification due to laser-induced microexplosions using time-resolved XRD technique, schematic principle (a), prototype of portable optical microscope system for practical dynamic XRD monitoring experiments.

optical microscope, which enables focusing of the optical pulses into the sample and *in situ* observation of the microexplosions, was prepared. This setup is shown in Fig. 2(b), with expected propagation of X-ray and optical beams indicated by the arrows. In the present setup, translation of the sample is achieved using simple manually-controlled mechanical translation stage, which is inconvenient for use. In the future studies the setup will be equipped with a transducer-controlled stage to facilitate automatic translation of the sample with each laser shot to facilitate exposure of pristine regions of the sample with each laser pulse.

2) 3D tomography XRD experiments on certain biological samples were performed aiming to visualize their internal structure. Nanoscale optical and structural properties of silk have been measured from 100-nm-thick longitudinal slices of silk fibers with  $\sim 10$  nm resolution. X-ray tomography and diffraction was used for structural characterization. Sub-wavelength resolution in



**Fig. 3.** X-ray tomography 3D rendered volume of a bunch of white silk Bombyx mori fibers at  $3.15 \mu\text{m}$  pixel resolution. The bunch was made of degummed single strand silk fibers.

hyperspectral mapping of absorbance and molecular orientation were carried out for comparison at IR wavelengths using synchrotron radiation. Reliable distinction of transmission by only 1-2% due to anisotropy of amide bands was obtained. Nanoscale composition of silk defining its unique properties via a hierarchical structural anisotropy can be analysed at the highest spatial resolution corresponding to the size of  $\beta$ -sheets, the crystalline building blocks of silk. Typical 3D image of a bunch of white silk Bombyx mori fibers is shown in Fig. 3. In addition, spectral characterization, lateral mapping and transmission with deep sub-wavelength resolution at IR molecular fingerprinting spectral window using thin 100 nm lateral slices of silk was performed. Absorbance and reflectance spectra of silk with resolution of SNOM tip (i.e. on the order of  $\sim 10 \text{ nm}$ ) were obtained. Absorbance from nano-thin silk slices with only 1.5% of the wavelength measured with a beam with lateral spot diameter comparable with the IR-wavelength were reliably measured and mapping across the silk fiber slice was obtained with a high fidelity. Orientational map at the absorbance bands was obtained. These studies have

demonstrated true nanoscale characterization of silk sample with nano-scale resolution in 3D. These results are currently under preparation for publication in a high-impact scientific journal.

### (3-2) Ripple effects and further developments

Exchange of scientific expertise between the participants was very helpful for coordination of experimental and theoretical work towards the goal of this project. Also, it has contributed to strengthening of domestic and international collaboration at Research Institute of Electronics. Exchange of scientific expertise between the participants was very helpful for coordination of experimental and theoretical work towards the goal of this project. Also, it has contributed to strengthening of domestic and international collaboration at Research Institute of Electronics. Joint work and discussions between the participants of this project have broadened the scope of existing domestic and international research collaborations at RIE, as well as opened new venues for collaboration in the future.

## [4] Achievements

List of publications:

- (1) I. Faniayeu, S.Khakhomov, I.Semchenko, V. Mizeikis, Highly transparent twist polarizer metasurface, Appl. Phys. Lett. 111, 111108 (2017).
- (2) Faniayeu, V. Mizeikis, Vertical split-ring resonator perfect absorber metamaterial for IR frequencies realized via femtosecond direct laser writing, Appl. Phys. Expr. 10 (2017).
- (3) V. Mizeikis, Direct laser writing of electromagnetic metasurfaces for infra-red frequency range (Invited talk), SPIE Photonics West 2018, San Francisco CA, Jan. 27-Feb. 1 (2018)
- (4) V. Mizeikis, Z. Hayran, H. Kurt, D. Gailevicius, M. Malinauskas, S. Juodkazias, K. Staliunas, Fabrication of optical field concentrators based on 3D chirped photonic crystals by direct laser writing technique, Meta 2017, Incheon, Korea, July 25-28 (2017).

## **Travelling report**

Name: Dr. Arturas Vailionis  
Affiliation: Stanford Nano Shared Facilities, Geballe Laboratory for Advanced Materials  
Period of time: 2017.11.27-12.03  
Destination: Shizuoka University, Japan  
Purpose: To carry out a joint research, plan the future collaboration, participate at the mini-workshop and report previously obtained results.  
Name of receiver: Prof. Vygantas Mizeikis

Name: Assoc. Prof. Akira Saito  
Affiliation: Division of Precision Sci. & Technology and Applied Physics, Graduate School of Engineering, Osaka University, Japan  
Period of time: 2017.11.28-29  
Destination: Shizuoka University, Japan  
Purpose: To carry out a joint research, plan the future collaboration, participate at the mini-workshop and report previously obtained results.  
Name of receiver: Prof. Vygantas Mizeikis