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IMAGING GRAINS IN A POLYCRYSTAL IN 3D WITH NANOACOUSTIC WAVES: SIGNAL PROCESSING METHODS.

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ABSTRACT

Picosecond laser ultrasonics methods provide non-contact, non-invasive imaging of μm samples, serving in biology to map cell mechanics, likewise in material science to scan microstructures. This work presents the 3D reconstruction of the cubic polycrystalline structure of ceria (CeO_2), with the time-domain Brillouin scattering (TDBS) method. We report on the development of signal processing methods to process the TDBS signals obtained by a scan of a micrometric area of ceria. 3D volumetric reconstruction of the grains is obtained with a maximum depth of 10 μm . Imaging contrast is enhanced thanks to the detection of quasi-shear acoustic waves, distinguishing small grains with slightly different crystalline orientations.

1. INTRODUCTION

Time-domain Brillouin scattering (TDBS) [1,2] is a pump-probe technique suited to the imaging of transparent materials like polycrystals [3]. In an opto-acoustic transducer (OAT), the pump fs laser pulse induces, via thermoelastic generation, an acoustic wave. When this wave is transmitted to the elastically anisotropic (cubic symmetry) ceria, up to three mode-converted acoustic pulses could propagate. The delayed probe fs laser pulse is strongly reflected at the fixed OAT surface, and weakly reflected at the propagating acoustic wavefronts in the optically isotropic grains of ceria. The interferences between these reflections produce, in the reflectivity signal collected by a photodetector, the so-called Brillouin oscillations which frequencies depend on the propagating acoustic pulses' velocities. Monitoring in time the Brillouin frequency changes of each acoustic mode, *i.e.* the velocity changes due to propagation in differently-oriented grains, give access up to three different views of the 1D distribution of the grains along the z-direction (depth). The 3D reconstruction is obtained by gathering 1D distribution from a 1 μm -step $60 \times 60 \mu\text{m}^2$ scan (xy-directions) of the sample, performed with an asynchronous optical sampling (ASOPS) system.

2. BRILLOUIN FREQUENCIES & PROCESSING

The three quasi acoustic modes that can be launched in ceria are: a quasi-longitudinal acoustic (LA) and two quasi-shear acoustic (TA) modes, labelled "slow" (STA) and "fast" (FTA). The velocity v of the acoustic wave propagating in the transparent sample are linked to the Brillouin oscillations frequency f_{BO} , at normal incidence of the probe beam, via the equation:

$$f_{BO} = \frac{2 n v}{\lambda}, \quad (1)$$

with n the refractive index of ceria and λ the probe laser wavelength. Estimation of the expected Brillouin frequency (BF) of these three possible acoustic modes are given in Table 1 using the elastic properties in Ref. [3] (Table 2).

Acoustic mode	Frequency interval (GHz)
LA	[58.72, 69.97]
STA	[26.77, 37.92]
FTA	[26.77, 42.45]

Table 1. Expected Brillouin frequency (BF) intervals to be detected in the case of cubic ceria (CeO_2) for each three acoustic modes.

Typical raw transient reflectivity signals measured via a photodetector are first subject to a filtering in order to remove the thermal background, as well as for de-noising. The 3600 acoustic signals obtained are subject to a signal-by-signal and mode by mode procedure in 2 steps: (i) the slicing and (ii) the shaping. The slicing consists in estimating the instantaneous BF of the signal in a short-time Hanning window. The strongest frequency component in the Fourier transform (FT) of the windowed acoustic signal is estimated by applying a synchronous detection technique (SDT) [4]. The instantaneous frequency is converted into instantaneous acoustic wave velocity using Eq. (1). Finally, each acoustic signal is converted into a vector containing the estimated BF with varying depth steps. Gathering these 3600 BF vectors leads to a final 4D (3D for spatial coordinates and 4D with the associated BF) sliced representation of the sample's grains. The shaping then

consists in a segmentation of the BF points forming the 4D sliced representation, using Otsu's image segmentation method [5]. The BF points that belongs to a common grain are aggregated in point clouds that are used as input of an alphashape algorithm [6], allowing a 3D volumetric reconstruction of the grains (Fig. 1).

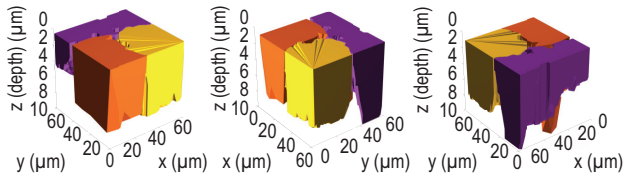


Figure 1. 3D reconstruction of the grains imaged with the LA mode contribution to the TDBS signal in three different viewing angles.

3. RESULTS & DISCUSSION

Each acoustic mode is differently sensitive to grain's orientation change. This imaging contrast effect is depicted in Fig. 2. The very first slice of the slicing is represented for each acoustic mode. Grains that appear as homogeneous when looking at the LA slice [Fig. 2(a)], are in fact composed of smaller grains only visible when processing the two TA modes BF intervals [Fig. 2(b-c)]. Thanks to this complementary information, a good agreement with the electron backscattered diffraction (EBSD) pattern [Fig. 2(d)] of the scanned area is demonstrated.

Reconstruction of the 3D grains obtained by the detection of TA modes not only agree with the grain boundaries shown by the LA mode, but brings also light on boundaries in the depth of the sample invisible to the LA mode. The ability of time-frequency tools to estimate the angle of an inclined buried interface between two differently oriented grains is one example of the wide range of information accessible from the presented results. Interestingly, grain boundaries can be identified via abrupt diminishing of the TDBS signals and by the BF shifting induced by the velocity change when the acoustic wave is transmitted from one grain to another.

4. CONCLUSION

The TDBS technique is applied on an interesting playground material, polycrystalline ceria, and signal processing methods are developed to reconstruct a 3D image of the sample's grains. For the first time, a complete 3D image of the scanned area is reconstructed, down to a depth of 10 μm and 3D grain boundaries are visualised. The contribution of the TA modes processing to the final 3D image highlights how crucial quasi-shear waves monitoring is for a complete characterisation of the sample. These results pave the way to TDBS imaging characterization, such as the analysis of buried inclined boundaries or else the determination of grain orientation. Being able to get such characteristics would bring major information on the roughness

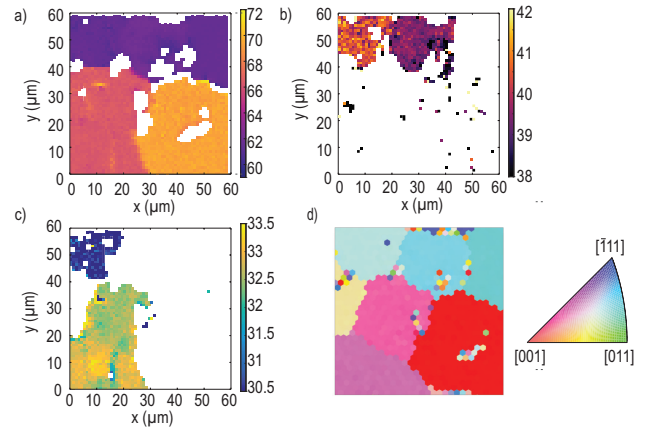


Figure 2. First slices obtained for the two types of acoustic mode: (a) LA mode, (b) TA mode (in the frequency interval [27, 37] GHz) and (c) TA mode (in the frequency interval [37, 44] GHz), (d) EBSD pattern of the scanned sample area.

of the grains interfaces that could explain macro-scale behaviours, such as ionic conductivity in the case of ceria.

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