Supporting information

Acoustic-shock wave-induced superheating-assisted dynamic recrystallization- a case-study of D-Tartaric acid

Sivakumar Aswathappa[1], Lidong Dai[1*] Sahaya Jude Dhas Sathiyadhas[2] and Raju Suresh Kumar[3]

[1]Key Laboratory of High-temperature and High-Pressure Study of the Earth's Interior, Institute of Geochemistry, Chinese Academy of Sciences, Guiyang, Guizhou 550081, China

[2]Saveetha School of Engineering, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu, 602105, India

[3]Department of Chemistry, College of Science, King Saud University, P.O. Box 2455, Riyadh 11451, Saudi Arabia

Corresponding author: dailidong@vip.gyig.ac.cn

Shock wave loading experiment

The Sigma Aldrich Company provided the commercially available D-Tartaric acid and the fine powder samples were purchased and utilized for the current study. For the experimental analysis, Mach number 4.7 shock waves (with a transient pressure and temperature of 16.5 MPa and 3175 K, respectively) were preferred such that two different sets of shock pulses with counts of 50 and 100 shocks were exposed on those two samples. The required shock waves have been generated by an in-house tabletop semiautomatic shock tube which has three sections known as driver, driven and diaphragm sections that are made of seamless steel. The driver and driven sections consist of long tubes of 48 cm and 180 cm, respectively and both have an inner diameter of 1.5 cm. Atmospheric air is used as the input source for the required shock wave generation. The diaphragm section separates the driver section and the driven section. Carbonless paper diaphragms are fed into the diaphragm section with the help of a motor. While the atmospheric air is being compressed into the driver section, at the critical pressure, the diaphragm is ruptured such that the shock wave is generated and moves along the driven section. For the shock loading, the sample pack with the dimension $10\times10\times1$ mm^3 are rigidly fixed in the sample holder which is fixed 1cm apart from the open end of the shock tube. Totally, 3 equal amount of samples have been chosen such that one sample has been kept as the control sample while the other sample has been treated with 250 and 500 shocks with the Mach number of 4.7. Fig. S1 (a and b) shows the schematic diagram of the experimental setup of the shock tube and in Fig. S2, the acoustic wave signals are presented which are recorded in the driven section. Subsequently, 50 and 100 shock pulses have been loaded on the test sample with an interval of 5 sec between each shock pulse. For example, 50 pulses mean shock wave-exposed on a sample 50 times with Mach number 4.7 (±0.1). For the present experiment, the shock waves of Mach number 4.7 with the reflected

transient pressure of 4.7 MPa (P_5) and the transient temperature of 3171 K (T_5) have been used and the values have been calculated by the standard R-H relations as in equations**.**

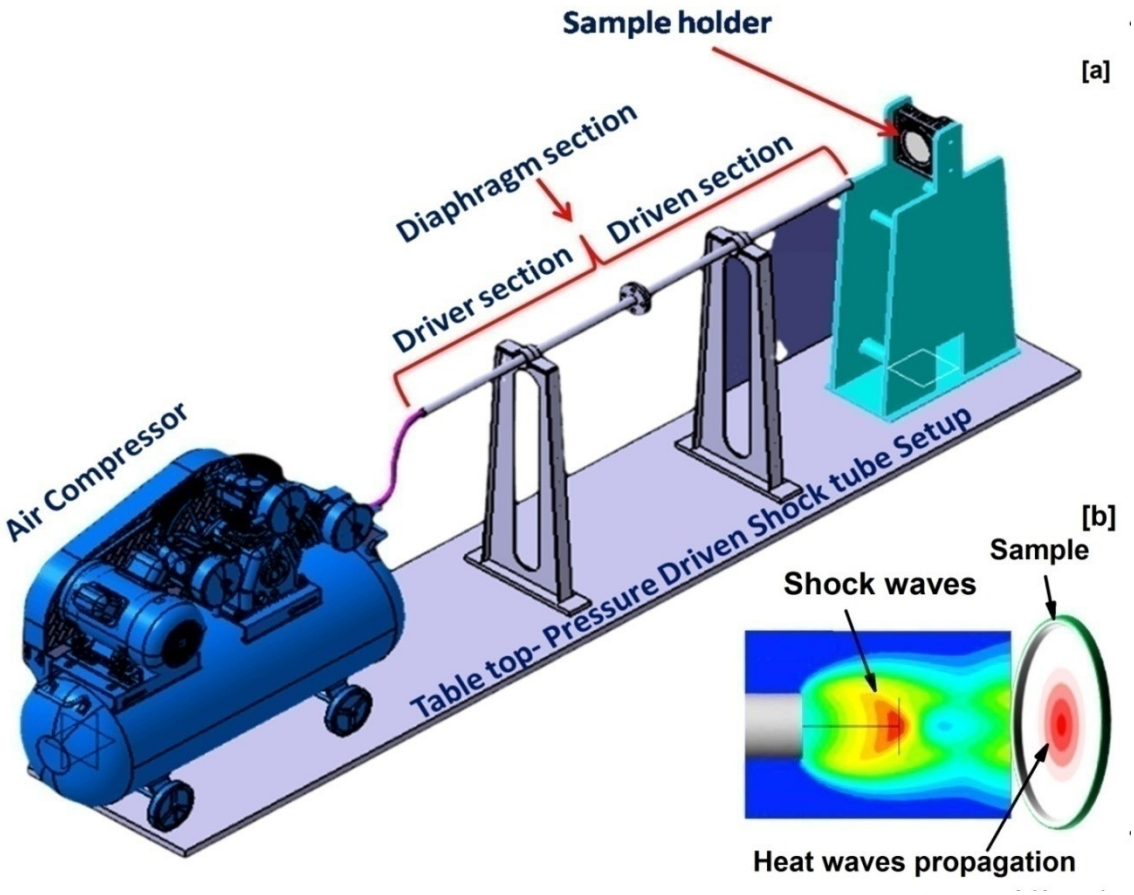

Fig. S1 (a) Schematic diagram of the table top pressure driven shock tube (b) shock propagation on the sample

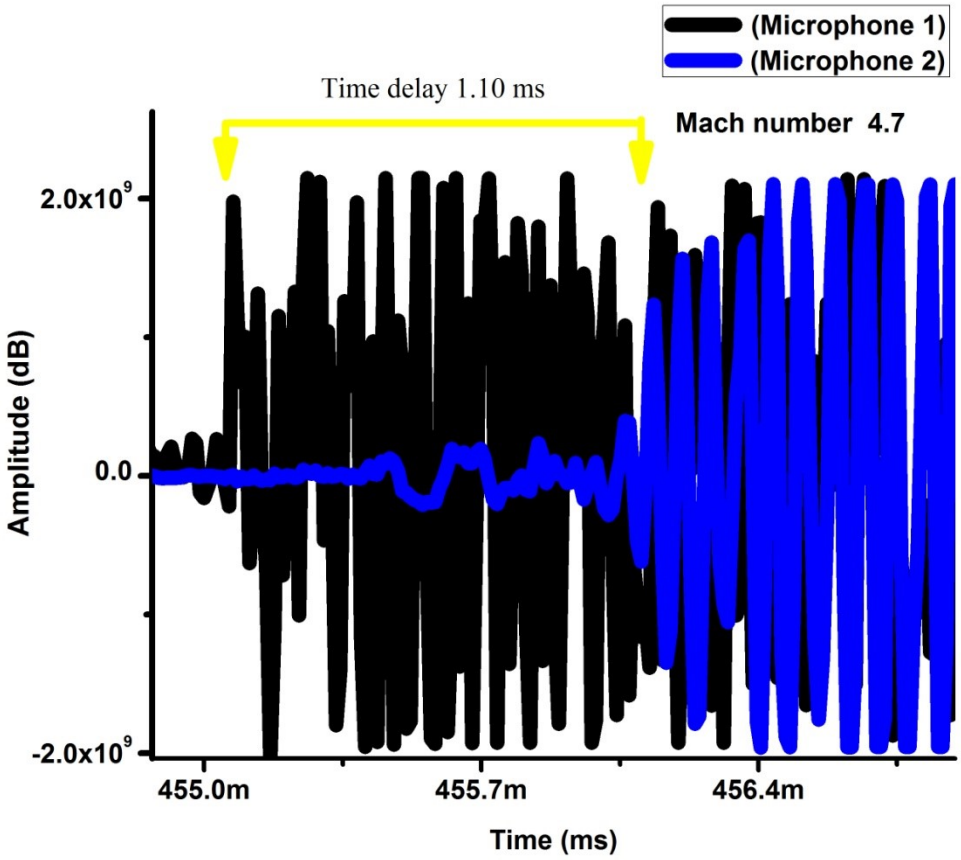

Fig. S2 Acoustic wave signals of the shock waves (recorded in the driven section)

R-H relations

$$P2 = P1\left[1 + \frac{2\gamma}{\gamma + 1}(M^2 - 1)\right] \text{..... (1)}$$

$$\frac{T_2}{T_1} = \frac{P_2}{P_1}\left[\frac{\left(\frac{\gamma + 1}{\gamma - 1}\right) + \frac{P_2}{P_1}}{1 + \left(\frac{\gamma + 1}{\gamma - 1}\right)\frac{P_2}{P_1}}\right] \text{..... (2)}$$

$$\frac{P_5}{P_2} = \frac{(3\gamma - 1)\frac{P_2}{P_1} - (\gamma - 1)}{(\gamma - 1)\frac{P_2}{P_1} + (\gamma + 1)} \quad \ldots\ldots\ldots (3)$$

$$\frac{T_5}{T_2} = \frac{P_5}{P_2}\left[\frac{\left(\frac{\gamma + 1}{\gamma - 1}\right) + \frac{P_5}{P_2}}{1 + \left(\frac{\gamma + 1}{\gamma - 1}\right)\frac{P_5}{P_2}}\right] \quad \ldots\ldots\ldots (4)$$

The initial fixed values are P_1 = 1 bar, γ =1.4 , T= 300 K, where M- Mach number, P_5 and T_5 are the reflected transient pressure and temperature at the end of the driven tube.

After the completion of the shock wave loading, the control and shock wave loaded samples have been analyzed by powder XRD, Raman, UV-DRS, XPS, FE-SEM, HR-TEM, VSM, DSC, TGA, electrical and electrochemical studies to understand the structural and functional property measurements such as optical, electrical and electrochemical properties.

Analytical experiments

Powder XRD

The analysis of Powder X-ray diffraction (PXRD) [Rigaku – Smart Lab X-Ray Diffractometer, Japan- CuKα1 as the X-ray source (λ = 1.5407 Å), with the step precision of ± 0.001°] was performed over the diffraction angle from 10-90 degree.

Raman Spectroscopy Experiment

We investigated the Raman spectra of the control and Tartaric acid using a Renishaw 2000 micro confocal Raman spectrometer coupled with a 532 nm argon ionic excitation source.

Single-crystal silicon with a characteristic Raman peak at 520.0 cm^{-1} was utilized to calibrate the Raman spectroscopy system prior to measurement. Raman spectra of the control and shocked Tartaric acid were gathered within the wavenumber range of 100−3500 cm^{-1} in the backscattering geometry with the spectral resolution of 1.0 cm^{-1} and the acquisition time was 120 s. The laser spot size was 50 μm^2 and the optical microscope's objective lens was used such that an X50 long working distance objective lens (WD = 10.6 mm) was utilized and the value of numerical aperture was 0.5 while the Raman data was collected by Renishaw Wire 5.1 instrument control and the data acquisition software. We processed the obtained Raman spectra with a Lorentzian-type function in Origin 9.0 software to extract the Raman peak position and its corresponding FWHM.

Scanning Electron Microscope (SEM)

Scanning Electron Microscope was used with (Carl Zeiss- Sigma 300), Resolution: 1.0 nm @ 15 kV, 1.6 nm @1kV Acceleration voltage: 0.02 kV to 30 kV. Magnification level was up to 10 lakhs times Smart EDX EDS analysis system and Probe current: 3pA – 20nA. Digital store with maximum resolution of 32768 x 24576 Pixel was utilized. Windows 10 (64 bit) has been used to understand the surface morphology of the control and shocked Tartaric acid samples.