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14. ABSTRACT
As a complement to real-time study of dynamic compression by in situ experiments and simulations, we will carry out recovery of shocked material and detailed characterization of the recovered products, in order to assess the possible formation of dense crystalline phases and (recoverable) changes upon impact in the structure of amorphous silicates, especially soda-lime glass.
• We will carry out new recovery experiments on isotopically-enriched starting glass, suitable for nuclear magnetic resonance characterization.
• We will analyze recovered powders supplied by ONR from experiments conducted elsewhere alongside our starting materials and new recovery samples.
• Both the existing and new samples will be studied by optical and electron microscopy, optical and X-ray Raman spectroscopy, X-ray diffraction, and NMR spectroscopy in order to characterize, at multiple scales from atomic-level upwards, the changes in structure and ordering after shock and recovery. The suite of analyses targets both bulk properties and element-selective properties and can be adapted to determine the local environment around O, Na, Al, Si, and Ca atoms.

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December 23, 2019

Re: Final Technical Report for ONR award Number N00014-17-1-2772
Title: Performance and advanced characterization of shock recovery experiments on soda-lime glass

Major Goals:

As a complement to real-time study of dynamic compression by in situ experiments and simulations, we will carry out recovery of shocked material and detailed characterization of the recovered products, in order to assess the possible formation of dense crystalline phases and (recoverable) changes upon impact in the structure of amorphous silicates, especially soda-lime glass.

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Accomplishments Under Goals:

• An isotopically enriched soda-lime glass was synthesized by our partners in Korea and delivered to Caltech.

• It was remelted to form it into a shape suitable for loading into our recovery chambers.

• A conventional steel-chamber reverberating recovery shot was carried out, with estimated peak pressure of 24 GPa in the sample.

• SEM, Electron probe, Electron Backscatter Diffraction, Raman spectroscopy, Focused Ion Beam milling, and TEM examination were all carried out on the recovered sample at Caltech.

• Magic-Angle Spinning (MAS) ^{27}Al NMR, ^{29}Si NMR, and Triple-Quantum-MAS ^{17}O NMR and ^{23}Na NMR characterization were carried out by our partners in Korea.

• Synchrotron micro-XRD and Oxygen K-edge inelastic X-ray scattering measurements were carried out on the recovery material at the Advanced Photon Source at Argonne National Laboratory.

• A powdered recovery material supplied by ONR was also characterized by several of these methods: SEM, EBSD, Raman, and synchrotron micro-XRD.

• The silicate portions of both samples (Caltech and ONR) are *entirely amorphous*. No crystalline silicate or oxide phases, whether low-pressure or high-pressure, were detected.

• Some metal phases were detected in the Caltech sample by synchrotron XRD, including FCC Fe-Cr-Ni alloy derived from the sample chamber and a nano-phase primitive-lattice Fe-Si alloy, formed by reaction at the chamber wall.

• O K-edge IXS on the Caltech sample indicates no measurable change in bond lengths or character from the point of view of O atoms in the glass.

• However, ^{27}Al NMR does show development of an increased population of octahedrally-coordinated Aluminum, one of the recognized mechanisms of permanent densification of alumino-silicate glasses. However, on the whole the dfferences between the starting materal and the recovered material are not significant. Conventional shock recovery on this material shows nearly complete annealing to the original low-pressure glass structure.

• In general, it appears that conventional shock reverberation experiments do not allow for recovery of high-pressure solids or significantly modified silica coordination in the bulk amorphous material. Hence, we repeated the recovery experiment with a different loading path. This was the first-ever attempt at a recovery experiment using a graded density impactor (GDI) to deliver a nearly isentropic ramp loading instead of a shock to an encapsulated sample. The idea is that an isentropically loaded and isentropically unloaded sample will recover to a near room-temperature condition, preventing the annealing to low-pressure structures that is typical of the hot release states from conventional shock loading.

• The experiment was a success, in that the chamber remained intact and sample was recovered. A split was sent to our collaborator in Korea, who has already collected preliminary ^{27}Al NMR data and is continuing with higher resolution data collection and study of the other NMR-active nuclei in the sample. The result is very encouraging ... there is clearly a shift in the coordination number statistics of the ^{27}Al compared to the starting material. This is the first time that high-pressure glass structure at the coordination number level has been preserved in a dynamically compressed glass! More characterization will follow.

• The samples sent by ONR for analysis are entirely amorphous. We detected no crystalline phases (such as stishovite) in this material, despite applying multiple sensitive analytical methods.

Training Opportunities:

A visiting scientist in our lab, Xiaojuan Ma, learned advanced shock wave experimental methods and theory of silicate glass compression on this project.

Postdoctoral scholar Jinping Hu took over the project after Ma left the lab and has added advanced shock wave methods and pyrometer to his toolkit. Given his interest in shocked meteorites and high-pressure mineralogy, this is important for his future career plans.

Graduate student Olivia Pardo, whose main project involves silicate glasses and liquids of geological interest, has observed our experiments on soda-lime glass as part of her preparation to do similar experiments on her own samples.

Results Dissemination:

Results have so-far been disseminated at the annual ONR-332 reviews in February 2018 and 2019 and at the biennial American Physical Society topical group meetings on Shock Compression in Condensed Matter in St. Louis and in Portland. A manuscript is still in preparation at this time, pending further (NSF-funded) experiments and calculations.

Follow-up plans:

A White Paper has been submitted to ONR describing potential next steps.

Sincerely,

Eleanor and John R. McMillan Professor of Geology and Geochemistry