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14. ABSTRACT

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON Valery Levitas
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RPPR Final Report

as of 10-Oct-2023

Agency Code: 21XD

Proposal Number: 77306TERIP

Agreement Number: W911NF-21-1-0313

INVESTIGATOR(S):

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Report Date: 14-Jun-2023

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Final Report for Period Beginning 15-Jun-2021 and Ending 14-Jun-2023

Title: System for Materials Study under High Pressure, Strain Rates, and Large Deformations

Begin Performance Period: 15-Jun-2021

End Performance Period: 14-Jun-2023

Report Term: 0-Other

Submitted By: Valery Levitas

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Distribution Statement: 1-Approved for public release; distribution is unlimited.

STEM Degrees: 0

STEM Participants: 3

Major Goals: The goal of the project is to develop an experimental setup including the dynamic Rotational Diamond Anvil Cell (dRDAC) device and measurement techniques to study the viscoplastic flow, phase transformations, and microstructure evolution in materials in a broad range of strain rates and couple it with our theoretical and computational efforts.

The current project will therefore involve the purchase of the items enumerated in the Table and installation of a complete dRDAC system including optical components at ISU. This new system will first be employed in routine dRDAC experiments at ISU while offering invaluable training to students and postdocs. Next, the system will also permit preliminary experiments at ISU in preparation for more extensive work at synchrotron facilities. Since the main component of the system, the RDAC, is portable it will easily be adapted for use at Sectors 13 - GSECARS and 16 - High Pressure Collaborative Access Team (HPCAT) at the Advanced Photon Source (APS) at the Argonne National Laboratory. In this way, dRDAC research will become more wide- spread nationwide. Until now, there no publications on the dRDAC-related experimental and theoretical research.

Accomplishments: Instead of one, two dynamic RDACs, for the same price were designed (with our contribution) and manufactured by Dr. Stas Sinogeikin (DAC Tools), a former beam scientist at HPCAT. The reason for such a gesture was that Dr. Sinogeikin believes that dRDAC has a great future, and he will get multiple orders for this device. The first dRDAC is for controlled rotation rates from very low to 10 RPM, and another one is for up to 4,000 RPM, with different design and engineering solutions. The first one was successfully tested in our laboratory at ISU and at HPCAT on Zr, Si, and olivine. We gave our feedback to Dr. Sinogeikin, which was taken into account in his design for the second dRDAC. The second dRDAC was manufactured and tested in our laboratory and integrated with new camera.

Training Opportunities: Two postdocs, Feng Lin and Sorb Yesudhas, worked on this project and participated in all activities, including search and ordering components, submitting orders, communicating with companies, installing devices and connecting them in the single system, testing, and performing experiments at ISU and HPCAT. They supervised three US undergraduate students (supported by NSF Research Experience for Undergraduate program), Michael Weber, Haley Schultz, and Ayman Karmi, who participated in most of the technical activities, including experiments at HPCAT. Two graduate students, Achyut Dhar and Raghunandan Pratoori, who are on internship at HPCAT (supported by NSF INTERN program and HPCAT directly) were trained by Feng Lin and Sorb Yesudhas.

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Results Dissemination: Results of the project obtained using rough diamonds within traditional DAC and RDAC and new dRDAC were disseminated through publications in highly ranked journals (some of them are open access journals) and preprints, presentations at conferences, invited seminars, and weekly meetings with HPCAT staff and my students, working as interns at HPCAT. Our papers, data, and some preprints are posted at the PI website at ISU, researchgate.net, and Purdue e-Pubs and Digital Repository @ ISU. Video recording of multiple PI's presentations at conferences and seminars are posted at <https://www.youtube.com/@valerylevitas8202/featured>.

Honors and Awards: Valery Levitas was elected to the EU Academy of Science (2022) and became a Fellow of the International Association of Advanced Materials (IAAM), Sweden (2023) and Murray Harpole Chair in Engineering (2023).

Paper "Levitas V.I. High-Pressure Phase Transformations under Severe Plastic Deformation by Torsion in Rotational Anvils. Material Transactions, 2019, Vol. 60, No. 7, 1294-1301" is recognized as the most cited paper in Material Transactions during 2016-2022.

Protocol Activity Status:

Technology Transfer: We are working together with the HPCAT Director Dr. Nenad Velisavljevic to make RDAC and dRDAC with traditional and rough diamonds available for the HPCAT general users. This goal is being taken into account during the current APS upgrade in terms of parameters of the measurement systems.

PARTICIPANTS:

Participant Type: Postdoctoral (scholar, fellow or other postdoctoral position)

Participant: Feng Lin

Person Months Worked: 12.00

Funding Support:

Project Contribution:

National Academy Member: N

Participant Type: Postdoctoral (scholar, fellow or other postdoctoral position)

Participant: Sorb Yesudhas

Person Months Worked: 12.00

Funding Support:

Project Contribution:

National Academy Member: N

Participant Type: Undergraduate Student

Participant: Michael Feber

Person Months Worked: 4.00

Funding Support:

Project Contribution:

National Academy Member: N

Participant Type: Undergraduate Student

Participant: Haley Schultz

Person Months Worked: 3.00

Funding Support:

Project Contribution:

National Academy Member: N

Participant Type: Undergraduate Student

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Participant: Ayman Karmi

Person Months Worked: 3.00

Project Contribution:

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Funding Support:

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Date Submitted: 10/5/23 12:00AM

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Publication Location: United States

Article Title: Recent In Situ Experimental and Theoretical Advances in Severe Plastic Deformations, Strain-Induced Phase Transformations, and Microstructure Evolution under High Pressure

Authors: Valery, Levitas

Keywords: strain-induced phase transformations, in situ experiments, rotational diamond anvils, high-pressure torsion, multiscale theory and simulations, microstructure evolution

Abstract: Severe plastic deformations (SPD) under high pressure, mostly by high-pressure torsion, are employed for producing nanostructured materials and stable or metastable high-pressure phases. However, they were studied postmortem after pressure release. Here, we review recent in situ experimental and theoretical studies of coupled SPD, strain-induced phase transformations (PTs), and microstructure evolution under high pressure obtained under compression in diamond anvil cell or compression and torsion in rotational diamond anvil cell. The utilization of x-ray diffraction with synchrotron radiation allows one to determine the radial distribution of volume fraction of phases, pressure, dislocation density, and crystallite size in each phase and find the main laws of their evolution and interaction. Coupling with the finite element simulations of the sample behavior allows the determination of fields of all components of the stress and plastic strain tensors and volume fraction of high-pressure

Distribution Statement: 1-Approved for public release; distribution is unlimited.

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Publication Type: Journal Article

Peer Reviewed: N

Publication Status: 5-Submitted

Journal: arXiv

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Publication Identifier: arXiv preprint arXiv:2307.11215

Volume:

Issue:

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Date Submitted: 10/5/23 12:00AM

Date Published:

Publication Location: United States

Article Title: Plastic strain-induced olivine-ringwoodite phase transformation at room temperature: main rules and the mechanism of the deep-focus earthquake

Authors: Feng, Lin, Valery, Levitas, Sorb, Yesudhas, Jesse, Smith

Keywords: Olivine-ringwoodite phase transformation, dynamic rotational diamond anvil cell

Abstract: Deep-focus earthquakes that occur at 350–660 km are theorized to be caused by strain-induced olivine-spinel phase transformation (PT). We introduce and apply dynamic rotational diamond anvil cell with rough diamond anvils to deform San Carlos olivine. While olivine was never transformed to spinel at any pressure at room temperature, we obtained olivine-ringwoodite PT under severe plastic shear at 15–28 GPa within seconds. This is conceptual proof of the difference between pressure- and plastic strain-induced PTs and that plastic straining can accelerate this PT from million years to timescales relevant for the earthquake. The PT pressure linearly reduces with increasing plastic strain, corresponding increasing dislocation density and decreasing crystallite size. The main rules of the coupled severe plastic flow, PT, and microstructure evolution are found.

Distribution Statement: 2-Distribution Limited to U.S. Government agencies only; report contains proprietary info

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Journal: Materials Research Letters

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Volume: 11

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Date Submitted: 10/5/23 12:00AM

Date Published: 7/12/23 5:00AM

Publication Location: United States

Article Title: In-situ study of rules of nanostructure evolution, severe plastic deformations, and friction under high pressure

Authors: Feng, Lin, Valery, Levitas, Krisha, Pandey, Sorb, Yesudhas, Changyong, Park

Keywords: High pressures; severe plastic deformations; in-situ synchrotron diffraction; yield strength; nanostructure evolution

Abstract: Severe plastic deformations under high pressure are used to produce nanostructured materials but were studied ex-situ. Rough diamond anvils are introduced to reach maximum friction equal to yield strength in shear and the first in-situ study of the evolution of the pressure-dependent yield strength and radial distribution of nanostructural parameters are performed for severely pre-deformed Zr. ?- Zr behaves like perfectly plastic, isotropic, and strain-path-independent and reaches steady values of the crystallite size and dislocation density, which are pressure-, strain- and strain-path-independent. However, steady states for ?-Zr obtained with smooth and rough anvils are different, causing major challenge in plasticity theory.

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Acknowledged Federal Support: Y

Publication Type: Journal Article Peer Reviewed: Y **Publication Status:** 5-Submitted

Journal: arXiv

Publication Identifier Type: DOI

Publication Identifier: 10.48550/arXiv.2305.15737

Volume:

Issue:

First Page #:

Date Submitted: 10/5/23 12:00AM

Date Published:

Publication Location: United States

Article Title: Rules of plastic strain-induced phase transformations and nanostructure evolution under high-pressure and severe plastic flow

Authors: Feng, Lin, Valery, Levitas, Krishan, Pandey, Sorb, Yesudhas, Changyong, Park

Keywords: Rough diamond anvils; plastic strain-induced phase transformations; nanostructure

Abstract: Severe plastic deformations under high pressure are used to produce nanostructured materials but were studied ex-situ. Rough diamond anvils are introduced to reach maximum friction equal to yield strength in shear and the first in-situ study of the evolution of the pressure-dependent yield strength and radial distribution of nanostructural parameters are performed for severely pre-deformed Zr. ?- Zr behaves like perfectly plastic, isotropic, and strain-path-independent and reaches steady values of the crystallite size and dislocation density, which are pressure-, strain- and strain-path-independent. However, steady states for ?-Zr obtained with smooth and rough anvils are different, causing major challenge in plasticity theory.

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Publication Status: 5-Submitted

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Publication Identifier: arXiv:2303.04407v

Volume: Issue:

First Page #:

Date Submitted: 10/5/23 12:00AM

Date Published:

Publication Location: United States

Article Title: Plastic strain-induced phase transformations in silicon: drastic reduction of transformation pressures, change in transformation sequence, and particle size effect

Authors: Sorb, Yesudhas, Valery, Levitas, Feng, Lin, K, Pandey, Jesse, Smith

Keywords: plastic strain-induced phase transformations; direct and inverse Hall-Petch effect

Abstract: Severe plastic deformations under high pressure are used to produce nanostructured materials but were studied ex-situ. Rough diamond anvils are introduced to reach maximum friction equal to yield strength in shear and the first in-situ study of the evolution of the pressure-dependent yield strength and radial distribution of nanostructural parameters are performed for severely pre-deformed Zr. α -Zr behaves like perfectly plastic, isotropic, and strain-path-independent and reaches steady values of the crystallite size and dislocation density, which are pressure-, strain- and strain-path-independent. However, steady states for β -Zr obtained with smooth and rough anvils are different, causing major challenge in plasticity theory.

Distribution Statement: 2-Distribution Limited to U.S. Government agencies only; report contains proprietary info
Acknowledged Federal Support: Y

Partners

I certify that the information in the report is complete and accurate:

Signature: Valery Levitas

Signature Date: 10/5/23 10:32PM

Final Report

Project title: Materials Study under High Pressure, Strain Rates, and Large Deformations

PI: Valery Levitas

Agreement No. W911NF2110313

Abstract

Experimental setup including the dynamic Rotational Diamond Anvil Cell (dRDAC) device and measurement techniques are developed to study the viscoplastic flow, phase transformations, and microstructure evolution in materials in a broad range of strain rates and couple it with our theoretical and computational efforts. The traditional and rotational diamond anvil cells (DAC and RDAC) and dRDAC with rough diamond anvils are introduced to reach maximum friction equal to the yield strength in shear. They intensify plastic flow, strain-induced PTs, and nanostructure evolution. All the experiments described below utilized rough-DA. Experiments DAC, RDAC, and dRDAC with rough diamond anvils were performed at Iowa State University and Sectors 16 - High Pressure Collaborative Access Team (HPCAT) at the Advanced Photon Source (APS) at the Argonne National Laboratory. Studies involved strain-induced phase transformations in Zr, Si, and olivine and revealed various new rules and quantitative results for each of these materials.

List of items acquired:

S. No.	Item details	Manufacturer / Supplier	Cost (\$)
1.	Custom rotational / torsional Diamond Anvil Cell (DAC) for low and high strain rates	DAC TOOLS LLC, NAPERVILLE, IL	33,500.00
2.	Slow motion setup and load monitoring system of RDAC	DAC TOOLS LLC, NAPERVILLE, IL	12,500.00
3.	PHANTOM V2512 high speed camera	Vision Research	53,000.00
4.	Laser Diode Source and Control Module	Laser Lab Source	1,980.00
5.	MY20X-804 20X Mitutoyo Plan-Apochromat Objective, 0.42 NA, 20.0 mm WD	Thorlabs. Inc.	2,293.39
6.	OCEAN-FX Spectrometer	Ocean Optics Inc.	5,671.00
7.	DAC seats	DAC TOOLS LLC, NAPERVILLE, IL	4,170.00
8.	Diamonds	Almax easyLab Inc.	10,859.68
9.	Optical components (Lenses, filters, mounts, mirrors and beam splitters)	ThorLabs, Edmund Optics and Newport	3,240.75
10.	Precision Ablation Laser Microfabrication	PALM-Scientific LLC	5,095.37
11.	Other miscellaneous items and alignment accessories	ThorLabs, Edmund Optics and Acme Tools	1,689.81
12.	Money used for purchasing optical components and camera from other sources	ThorLabs, Vision Research	13,000.00
13.	Total		155,500*

*Grant funding is \$134,000. The difference was paid from other sources available to the PI.

Goal:

The goal of the project is to develop an experimental setup including the dynamic Rotational Diamond Anvil Cell (dRDAC) device and measurement techniques to study the viscoplastic flow, phase transformations, and microstructure evolution in materials in a broad range of strain rates and couple it with our theoretical and computational efforts.

The current project will therefore involve the purchase of the items enumerated in the Table and installation of a complete dRDAC system including optical components at ISU. This new system

will first be employed in routine dRDAC experiments at ISU while offering invaluable training to students and postdocs. Next, the system will also permit preliminary experiments at ISU in preparation for more extensive work at synchrotron facilities. Since the main component of the system, the RDAC, is portable it will easily be adapted for use at Sectors 13 - GSECARS and 16 - High Pressure Collaborative Access Team (HPCAT) at the Advanced Photon Source (APS) at the Argonne National Laboratory. In this way, dRDAC research will become more wide- spread nationwide. Until now, there no publications on the dRDAC-related experimental and theoretical research.

Accomplishments:

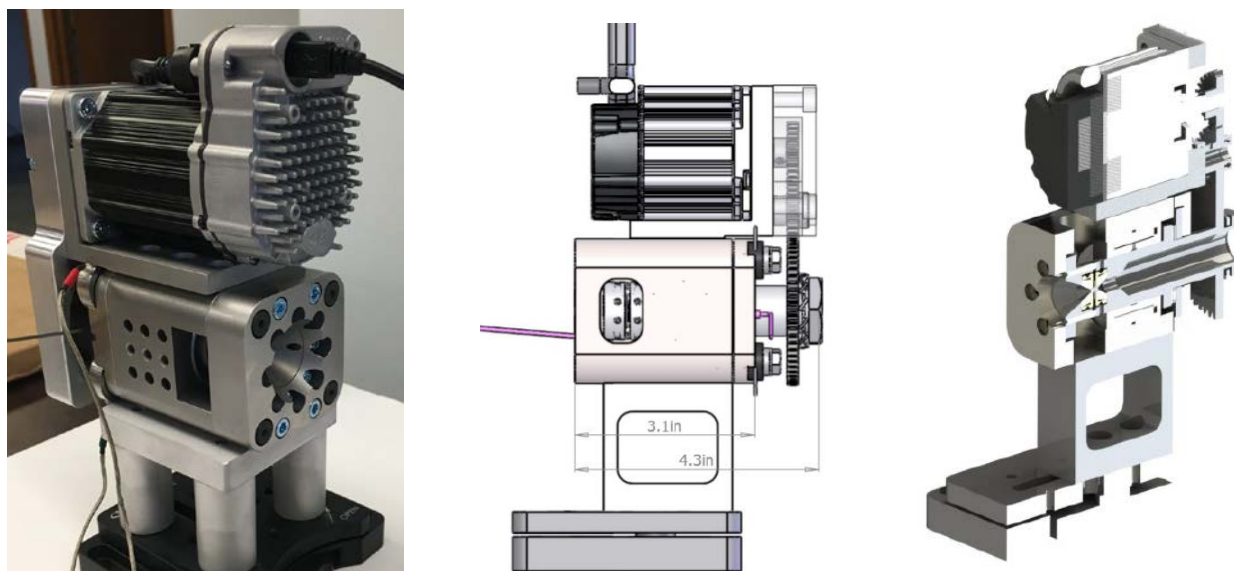


Fig. 1. Picture and schematics of dRDAC

Instead of one, two dynamic RDACs, for the same price were designed (with our contribution) and manufactured by Dr. Stas Sinogeikin (DAC Tools), a former beam scientist at HPCAT. The reason for such a gesture was that Dr. Sinogeikin believes that dRDAC has a great future, and he will get multiple orders for this device. The first dRDAC is for controlled rotation rates from very low to 10 RPM, and another one is for up to 4,000 RPM, with different design and engineering solutions. The first one was successfully tested in our laboratory at ISU and at HPCAT on Zr, Si, and olivine. We gave our feedback to Dr. Sinogeikin, which was taken into account in his design for the second dRDAC. The second dRDAC was manufactured and tested in our laboratory and integrated with a new camera.

1. DAC, RDAC, and dRDAC with rough diamond anvils (rough-DA) [1-5]

We introduced the DAC, RDAC, and dRDAC with rough diamond anvils (rough-DA) (Fig. 2) to reach maximum friction equal to the yield strength in shear. They intensify plastic flow, strain-induced PTs, and nanostructure evolution. All the experiments described below utilized rough-DA. The method was developed and the pressure-dependence of the yield strength of ω -Zr was determined. The rough-DA allowed us also to solve several other basic problems and brought up discoveries described in Sections 2-5. In addition, rough-DA eliminates the problem of describing contact friction required for modeling deformational and PT processes in DAC and RDAC. For traditional high-pressure torsion with ceramic/metallic anvils, friction reaches the maximum possible level due to large asperities. Utilizing rough-DA in RDAC will allow in situ studies of high-pressure torsion. Also, to increase the maximum possible pressure in DAC, toroidal grooves are used, which increase friction. This can be done with rough-DA more uniformly throughout the culet and with smaller stress concentrators.

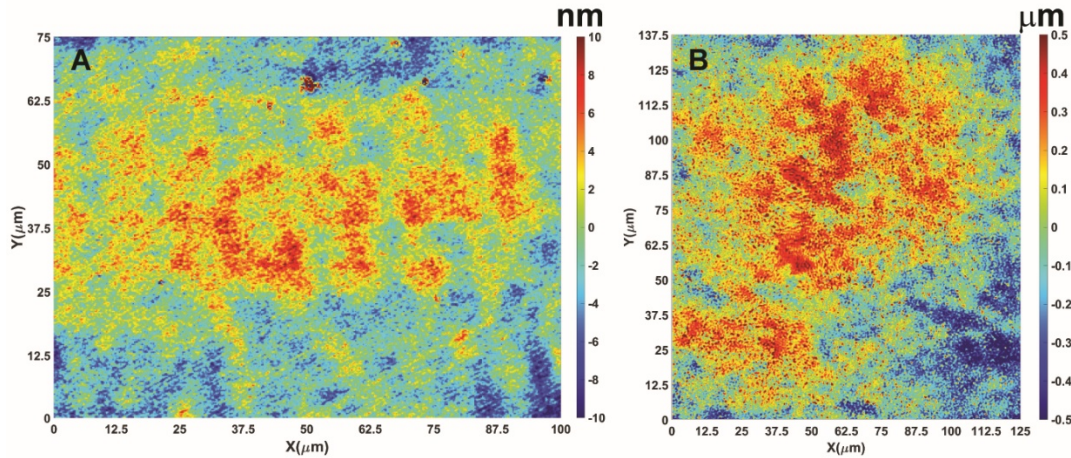


Fig. 2. Surface asperity profile of a smooth anvil and a rough-DA. (A) a traditional smooth diamond anvil with a range [-10 nm; 10 nm] and (B) a rough-polished diamond anvil (rough-DA) with a range [-500 nm; 500 nm].

2. New rules of high-pressure phase and nanostructure evolution and severe plastic flow [2,3,5]

The main problem in studying plasticity, plastic strain-induced PTs, structural changes, and contact friction is that they depend on five components of the plastic strain tensor ϵ_p and its entire path ϵ_p^{path} , making an unspecifiable number of combinations of independent parameters. This leaves no hope of fully comprehending and characterizing. We applied rough-DA to compression of severely pre-deformed Zr, because Zr PT behavior is much simpler than that for Si, with only one α - ω PT below 30 GPa. For the first time, we determined in situ not only distribution of pressure in phases and volume fraction of a high-pressure phase (what we did in 2020 for the first time),

but also crystallite size d and dislocation density ρ in phases. The first general experimental rules were drafted as follows:

(a) The minimum pressure for plastic strain-induced α - ω PT in Zr p_ε^d , the pressure-dependent yield strength $\sigma_y(p)$ of ω -Zr, crystallite size d_ω , and dislocation density ρ_ω are getting steady and independent of plastic strain tensor $\boldsymbol{\varepsilon}_p$ and its entire path $\boldsymbol{\varepsilon}_p^{path}$! Also, d_ω , and ρ_ω are getting independent of pressure.

(b) ω -Zr behaves like perfectly plastic, isotropic, and strain path-independent.

(c) Crystallite size d_ω and dislocation density ρ_ω evolution in ω -Zr during α - ω PT depends solely on the volume fraction c of ω -Zr and is independent of $\boldsymbol{\varepsilon}_p$, $\boldsymbol{\varepsilon}_p^{path}$, p , initial d_α , and anvil asperities.

(d) The kinetics of strain-induced PT depends not only on plastic strain but also on time, in contrast to all previous publications. That means that stress-induced PT at fixed $\boldsymbol{\varepsilon}_p$ occurs simultaneously with plastic strain-induced PTs, and new experimental and theoretical treatments are necessary.

(e) Rough-DA changes the plastic-strain part of the kinetics from the first order (typical for multiple nucleation and growth within the parent phase) to zero order (typical for propagation from a limited number of nuclei without their interaction, like for thickening of PT band).

(f) Rough-DA produce a different steady state and microstructures than the traditional diamonds for α -Zr, with smaller crystallite size and larger dislocation density, which results in smaller $p_\varepsilon^d = 0.67$ GPa (vs. 1.36 GPa for smooth anvils, i.e., 9 times lower than for hydrostatic loading).

The obtained results significantly enrich the fundamental understanding of plasticity, PTs, and nanostructure, and create new opportunities in material design, synthesis, and processing of nanostructured materials by coupling severe plastic deformations and PT at low pressure. We plan to obtain similar results for Si, which is much more complex due to multiple PTs.

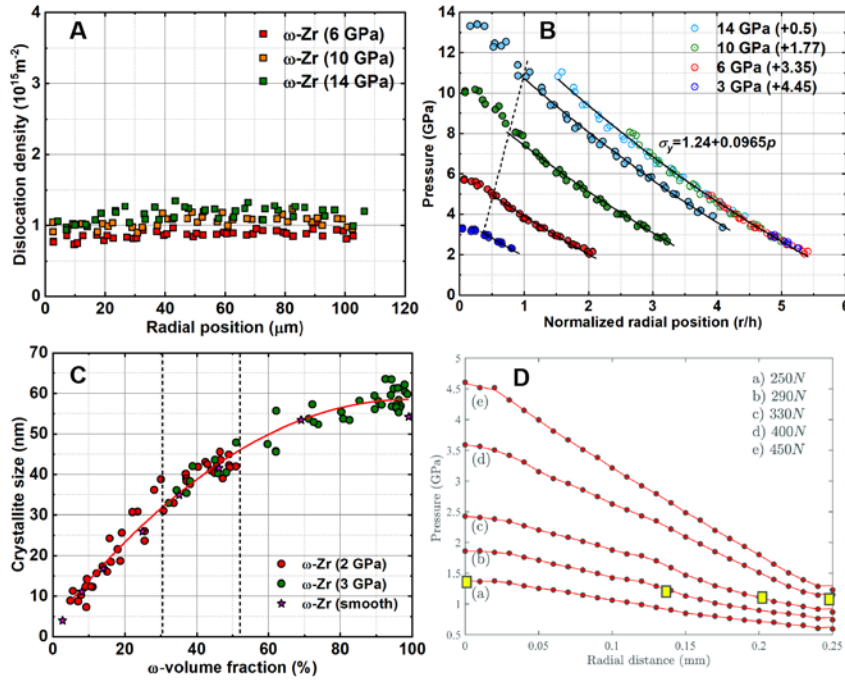


Fig. 3. Radial distributions of the dislocation density (A) and pressure (B, D) in DAC with rough-DA; the crystallite size (C) vs. volume fraction of ω -Zr. Squares in (D) correspond to the minimum pressure for plastic strain-induced α - ω PT in Zr p_{ε}^d , which is independent of radius, and consequently of plastic strain tensor ε_p and its entire path ε_p^{path} .

3. Plastic strain-induced phase transformations in silicon under non-hydrostatic compression and torsion: in situ x-ray study [4]

Hydrostatic Compression

Micron Si: I $\xrightarrow{13.5 \text{ GPa}}$ I+II+XI $\xrightarrow{15.3 \text{ GPa}}$ XI $\xrightarrow{16.5 \text{ GPa}}$ V $\xrightarrow{0 \text{ GPa}}$ XII+III (PTM: He)

100 nm Si: I $\xrightarrow{16.2 \text{ GPa}}$ I+II+XI $\xrightarrow{18.1 \text{ GPa}}$ XI $\xrightarrow{19.3 \text{ GPa}}$ V $\xrightarrow{0 \text{ GPa}}$ XII+III (PTM: He)

Non-hydrostatic Compression

Micron Si: I $\xrightarrow{2.5 \text{ GPa}}$ I+II $\xrightarrow{9.5 \text{ GPa}}$ I+II+XI $\xrightarrow{15 \text{ GPa}}$ XI $\xrightarrow{15.2 \text{ GPa}}$ V $\xrightarrow{0 \text{ GPa}}$ XII+III+a (S.S.)

100 nm Si: I $\xrightarrow{0.4 \text{ GPa}}$ I+II $\xrightarrow{8.9 \text{ GPa}}$ I+II+XI $\xrightarrow{14.7 \text{ GPa}}$ XI $\xrightarrow{15 \text{ GPa}}$ V $\xrightarrow{0 \text{ GPa}}$ XII+III (Cu)

Torsion

100 nm Si: I $\xrightarrow{5^\circ, 0.4 \text{ GPa}}$ I+II $\xrightarrow{5^\circ, 0.6 \text{ GPa}}$ I+II+III $\xrightarrow{82^\circ, 8.3 \text{ GPa}}$ I+II+III+XI $\xrightarrow{0 \text{ GPa}}$ XII+III (Cu)

100 nm Si: I $\xrightarrow{2.2 \text{ GPa}}$ I+II $\xrightarrow{31.8^\circ, 4.2 \text{ GPa}}$ I+II+III $\xrightarrow{0 \text{ GPa}}$ I+II&I+III&I+II+III Rough anvils (S.S.)

100 nm Si: I $\xrightarrow{9.1^\circ, 3.6 \text{ GPa}}$ I+II $\xrightarrow{0 \text{ GPa}}$ I Rough anvils (S.S.)

Fig. 4. Coexisting Si phases under different loadings/unloadings up to 20 GPa. PMT means pressure-transmitting medium, S.S. is for stainless steel gasket, 0 GPa corresponds to full unloading. Different results are separated by & are for different regions of the same sample.

All experiments have been performed at Advanced Photon Sources at Argonne National Laboratory. The first in situ studies of strain-induced phase transformations (PTs) in Si under compression in diamond anvil cell (DAC) (during COVID, DAC could be used only) and torsion in rotational DAC (RDAC) were performed. Our breakthrough results are summarized in Fig. 4:

- For nano-Si, plastic straining reduces Si-I \rightarrow II PT pressure from 16.2 GPa (hydrostatic) to 0.4 GPa, i.e., by a factor of 40.5! This pressure is also lower by a factor of 26.3 than the phase equilibrium pressure! For micron-Si, the pressure reduction for this PT is from 13.5 to 2.5 GPa.
- For PT I \rightarrow II, the minimum pressure for strain-induced PT $p_{\epsilon}^d = 0.4$ GPa in 100 nm Si is the same for compression in DAC and torsion in RDAC. Since the plastic strain tensor and its path are very different for compression and torsion, this means that p_{ϵ}^d is independent of the plastic strain tensor and its path. This rule reveals that the physics of strain-induced PTs under compression in DAC and torsion in RDAC do not differ fundamentally, and strain-induced PTs can also be studied in DAC. This discovery was very timely and allowed us to perform work on the project during COVID time because remote online experiments with traditional DAC were possible only. However, RDAC has a strong advantage in allowing various controllable pressure-shear loading programs, particularly at constant pressure, close to p_{ϵ}^d , whereas in DAC, pressure significantly grows during compression. This is crucial for not only initiating but also completing the desired PTs at low pressure.
- Under the plastic shear of 100 nm Si in RDAC, Si-III initiates at 0.6 GPa, while it does not appear under hydrostatic compression.
- For 100 nm Si, under torsion, PT occurs in the sequence Si-I \rightarrow I+II \rightarrow I+II+III, in contrast to different PT sequences suggested in ex-situ study in the previous literature.
- The coexistence of four phases, Si-I, Si-II, Si-III, and Si-XI, under torsion of 100 nm Si was observed for the first time.
- Based on gained understanding, we resolved long-standing puzzles, namely, to retain Si-II at ambient pressure and obtain reverse Si-II \rightarrow Si-I PT, demonstrating the possibilities of manipulating different synthetic paths. Retaining a large amount of Si-II will allow us to study it with traditional *ex-situ* methods (SEM, TEM, mechanical properties, etc.).
- We recovered single-phase nano-Si-III when unloading Si-V for 100 nm Si subjected to plastic compression in DAC, which was never done before. Nano Si-III has potential applications in electronics but was obtained by high-pressure torsion at 24 GPa and 10 anvil turns.

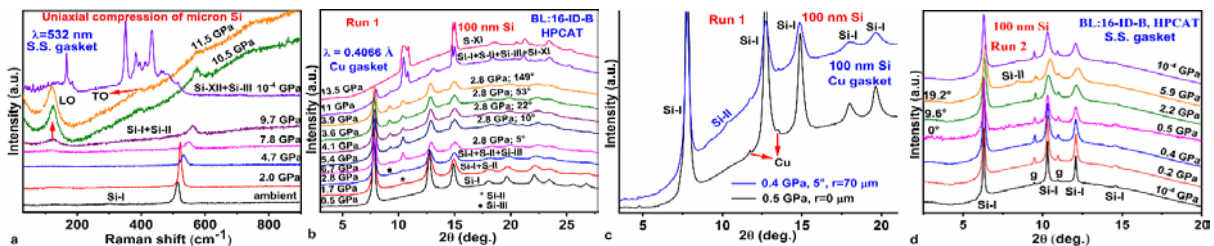


Fig. 5. Examples of in situ experimental studies of Si in DAC/RDAC. (a) Raman spectra for compression of micron-Si. (b) XRD study of PTs in nano-Si under compression and torsion in RDAC, including coexistence of 3 and 4 phases. (c) XRD detection of appearance of Si-II at 0.4 GPa and 5° rotation in nano-Si. (d) XRD monitoring of the loading in RDAC leading to the reverse PT Si-II→Si-I in nano-Si.

4. Particle size effect on PT in Si [4]

According to our previous theory of strain-induced nucleation at the dislocation pileup, the greater the grain size is, the stronger the stress concentration and consequently, the reduction in PT pressure. However, this is opposite to what we found in experiments, for α - ω PT in Zr. To eliminate this contradiction, we utilized our phase field, molecular dynamics, and concurrent atomistic-continuum simulations. In contrast to the previous analytical solution utilized in, dislocation pileup length is not related to the grain size since most dislocations are localized at the grain boundary producing a step (superdislocation). New theory is developed which correlates the grain-size dependence of the yield strength (Hall-Petch relationship) and the minimum pressure for strain-induced PT p_{ε}^d . Our theoretical predictions on grain size dependence of the minimum pressure for strain-induced PT p_{ε}^d are confirmed for the appearance of Si-II, i.e., there is a decrease in p_{ε}^d from micron to 100 nm particles. This is opposite to what we found in experiments under hydrostatic loading. For the next PTs, to Si-XI and V, this is not expected because the actual grain size after previous PT(s) is unknown and may not be related to the initial particle size; also, PT occurs in a mixture of phases instead of single phase, and phase interfaces may serve as additional obstacles for dislocation pileup. Determination of grain size evolution that we plan in future work will allow us to shed light on this problem.

5. Plastic strain-induced olivine-ringwoodite phase transformation at room temperature: main rules and the mechanism of the deep-focus earthquake [3]

Deep-focus earthquakes that occur at 350–660 km are theorized to be caused by strain-induced olivine→spinel phase transformation (PT). We introduce and apply dynamic rotational diamond anvil cell with rough diamond anvils to deform San Carlos olivine. While olivine was never transformed to spinel at any pressure at room temperature, we obtained olivine-ringwoodite PT under severe plastic shear in dRDAC at 15-28 GPa within seconds. This is conceptual proof of the difference between pressure- and plastic strain-induced PTs and that plastic straining can accelerate this PT from million years to timescales relevant for the earthquake. The PT pressure linearly reduces with increasing plastic strain, corresponding increasing dislocation density and

decreasing crystallite size. The main rules of the coupled severe plastic flow, PT, and microstructure evolution are found.

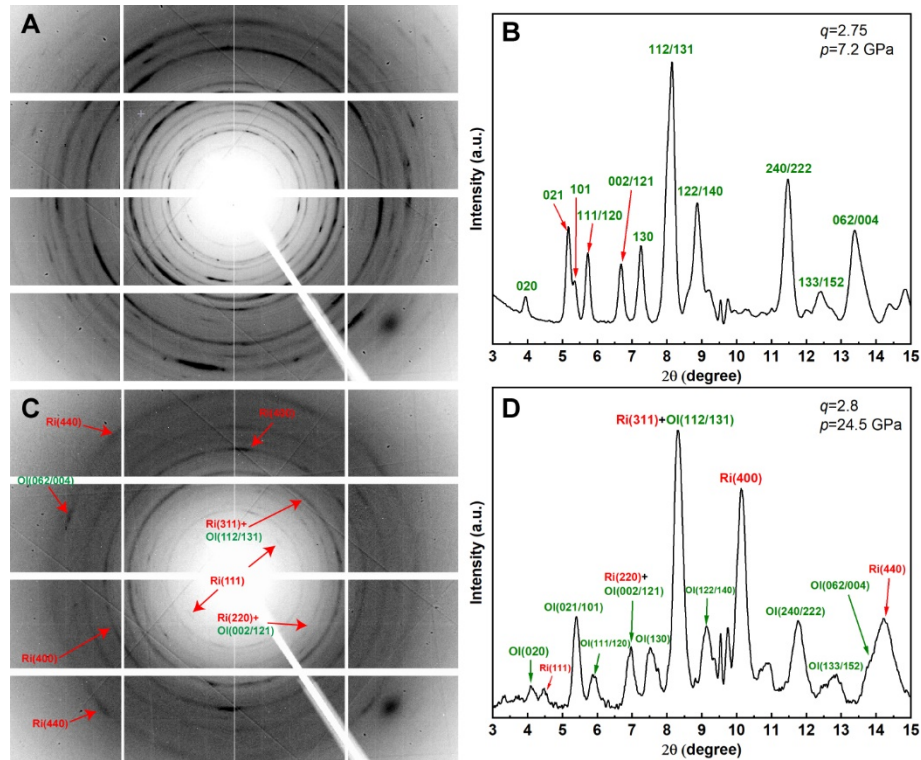


Fig. 6. X-ray diffraction patterns of deformed olivine sample. (A) 2D patterns and **(B)** integrated peak profiles after olivine reaching $p=7.2$ GPa at the sample center and 180° of anvil rotation. **(C)** 2D patterns and **(D)** integrated peak profiles of olivine and ringwoodite after reaching $p=24.5$ GPa at the sample center and 270° of anvil rotation.

References

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