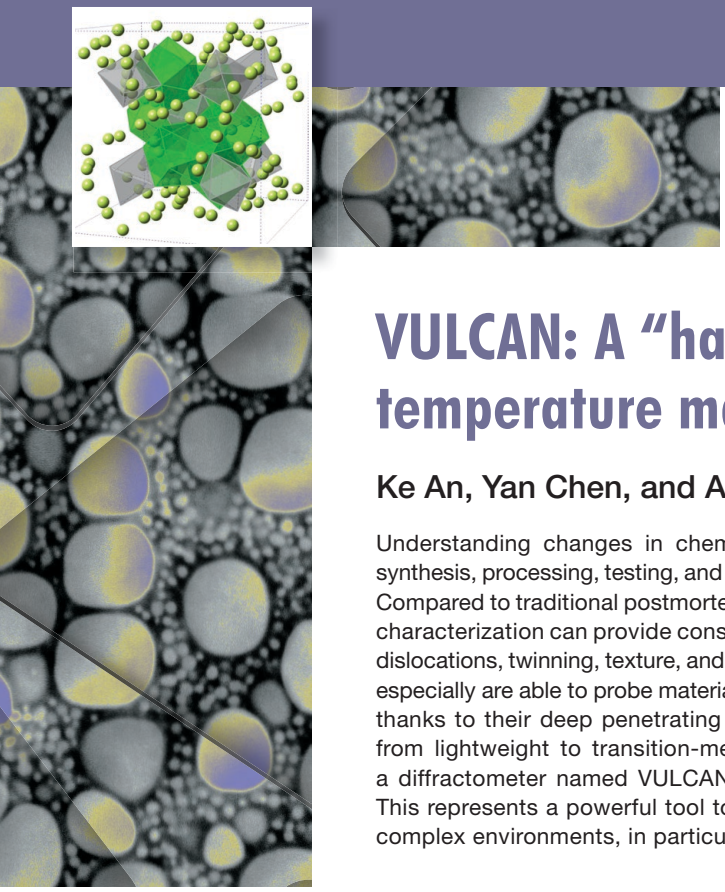


VULCAN: A "hammer" for high-temperature materials research

Ke An, Yan Chen, and Alexandru D. Stoica

Understanding changes in chemistry, microstructure, and physical properties during synthesis, processing, testing, and even service is vital for materials design and performance. Compared to traditional postmortem material characterization tools, *in situ* crystallographic characterization can provide considerable data and information on evolution of chemistry, dislocations, twinning, texture, and strains when a material is under external stimuli. Neutrons especially are able to probe material bulk properties and behaviors in extreme environments, thanks to their deep penetrating power and unique sensitivity to differentiate elements from lightweight to transition-metal atoms. In this article, we introduce and describe a diffractometer named VULCAN, which is located at Oak Ridge National Laboratory. This represents a powerful tool to understand materials properties and behaviors under complex environments, in particular, at high temperatures.

Introduction

High temperature is an environment that many functional and structural materials, including ceramics, oxides, and metals, undergo during synthesis, processing, testing, and service. Often, one or more other environments are combined with high temperatures, such as mechanical, corrosive, magnetic, electric, and radioactive environments. Under such extreme stimuli/perturbations, both crystallographic and morphological changes occur, such as phase transformation, ordering and disordering transition, magnetic transition, recrystallization, coarsening, phase segregation, precipitation, slip, dislocation, twinning, grain reorientation, and straining.

Having an understanding of materials properties and behaviors in such environments is vital for materials design, performance, reliability, and efficiency in terms of cost, environment, and safety. Recent advances in modern instrumentation and methodologies enable unprecedented capabilities in both spatial and temporal scales. Compared to traditional postmortem tools that provide static and limited data of material changes, *in situ* crystallographic characterization of materials yields time- and spatial-resolved data and information when materials are under external stimuli. This leads to a better understanding of scientific phenomena that are process-, condition-, and time-dependent.

Neutrons, compared to electrons and photons, have several unique advantages for probing material bulk properties and behaviors within extreme environments. A neutron scatters by interaction with nuclei, and its elemental scattering power is independent of elemental periodic order. It has great sensitivity to lightweight elements and is able to differentiate elements with near-atomic position such as transition metals, and even isotopes. Neutrons are heavy and penetrating compared to electrons and x-rays, allowing for the easy implementation of complex sample environments. Neutrons are also sensitive to magnetism. Although neutron flux is relatively low compared to synchrotron sources, modern neutron sources such as the Spallation Neutron Source (SNS) at Oak Ridge National Laboratory (ORNL), the world's most intense pulsed neutron source, have been able to produce much higher neutron brightness with an increase of several orders of magnitude than prior generation sources. With the high flux neutrons, advanced instrumentation, and complex sample environments, neutron instruments have become one of the most powerful modern tools for materials with great impact in both fundamental research and applied development.

The VULCAN instrument

The VULCAN instrument[1,2] at SNS is a time-of-flight materials science and engineering diffractometer. As schematically shown in **Figure 1**a, the motorized incident slits allow selectable apertures for the incident neutron beam. It is equipped

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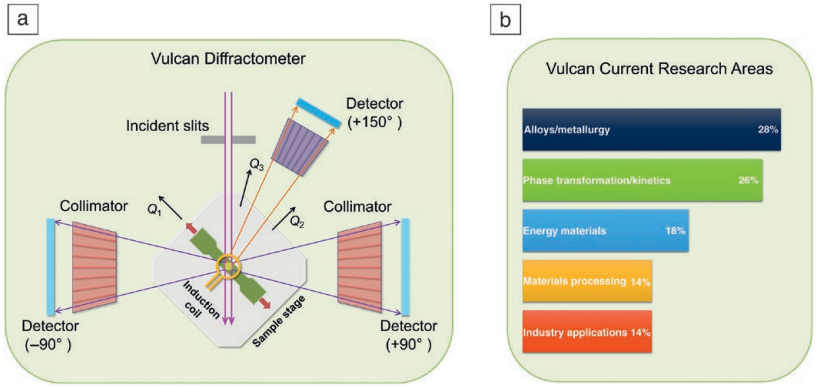


Figure 1. The setup and research on VULCAN. (a) A schematic of the experimental layout (Q_1, Q_2, and Q_3, are the scattering vectors for –90°, +90°, and +150° detectors, respectively), and (b) current research areas from user community proposals.

currently with three two-dimensional detector banks, located at +/–90° and 150° off the incident neutron beam and 2 m from the sample position. A gauge volume (ranges from 1–600 mm^3) inside a large structure under different sample environments is defined by the intersection of the incident beam and the field of view of radial collimators in front of the detector banks. The detector banks measure different directional crystallographic information along the bisector of the incident beam and the scattering direction of each detector.

The sample stage supports robust sample environments and engineering structures for *in situ* characterization and mapping scans. Various sample environments with individual or combined fields can be mounted on VULCAN for *in situ* measurements. For example, high-temperature standalone furnaces with either a controlled atmosphere or vacuum could enable heating up to 1600°C. The VULCAN load frame and auxiliary induction heating coils enable thermomechanical testing/processing. A resistive heating gas enclosure load frame furnace[3] has been developed with conductive heating rates of up to 600°C/s. We have also demonstrated the use of neutron electrostatic levitation facility for high-temperature liquids.[4]

In the SNS, the acquired neutron data are registered by the time-event mode,[5] where individual neutrons are time-stamped, and VULCAN often operates in a real-time continuous neutron data collection mode during *in situ* parametric studies. Data chopping/slicing by different time bins can be performed by using VDRIVE[6] (VULCAN Data Reduction and Interactive Visualization softwarE) based on required data statistics and synchronized with sample environment parameters. The time bin for a diffraction pattern spectrum with sufficient statistics depends on the gauge volume, size of samples, and elemental compositions as well as crystallographic structures. For common Fe- or Ni-based alloys, typical measurement time with ~250 mm^3 gauge volume can be as short as 1 s for phase transformation studies, which is comparable to high-energy synchrotron x-rays. The time interval can be reduced to milli- or microseconds by using a larger beam size or stroboscopic approaches,[3] respectively.

VULCAN enables *in situ* crystallographic understanding or characterization of phenomena such as phase transformation,[7–9] site elemental occupancies,[10,11] atomic-level ordering or disordering,[12–14] kinetics of recrystallization,[15] elastic anisotropy,[16,17] texture,[18,19] elastoplastic load sharing,[20] stacking-fault probability[21,22] and twinning,[21,23–26] strengthening[20] and hardening,[27] plasticity mechanisms,[28,29] shape-memory effects,[30–34] piezoelectric effects,[35] and batteries[36–38] under external stimuli. As shown in Figure 1b, current research covers diverse science and engineering fields, where metallurgy and phase transformation/kinetics are the lead areas that account for more than one-half of the total conducted research. In addition, VULCAN has attracted the engagement of industry in using neutrons. While VULCAN is capable of tackling various problems, here we specifically highlight its scientific high-temperature capabilities in studies of functional materials and structural alloys, as well as additive manufacturing (AM).

High-temperature solid-state synthesis and processing

High-temperature solid-state synthesis or thermal processing can involve reversible, simultaneous, and complex processes among multiple species of reactants that are hard to monitor. The thermal treatment that controls chemical transportation and structural transition tunes materials performance, and thermal stability is also of importance for material applications. *In situ* high-temperature neutron diffraction (HTND) is capable of differentiating between multiple phases and evolutions, thus offering a direct tool to reveal the synthesis mechanism and aid the process optimization for producing target compounds.

Figure 2[39] shows an example where by tracking the reaction and kinetics among multiple (up to nine) phases in the synthesis of the Li-ion solid electrolyte $Li_7La_3Zr_2O_{12}$ (LLZO), HTND revealed the appearance of essential intermediate phases before the final cubic LLZO was formed. Minor dopants can thus be utilized to alter the intermediate phases and reaction kinetics, in order to stabilize the cubic LLZO upon cooling. In addition, the natural sensitivity of neutrons to light elements offers a unique capability of understanding the mechanism at the atomic scale and reveals how Li vacancies were distributed in the doped LLZO. It was discovered that not all vacancies contribute to Li transportation in the solid; only the active vacancy density determines the three-dimensional (3D) Li conduction pathway, which can be tuned by engineering the dopant valence and concentration.[10]

Furthermore, neutrons can differentiate between transition metals and isotopes. For example, the $LiNi_{0.5}Mn_{1.5}O_4$ spinel

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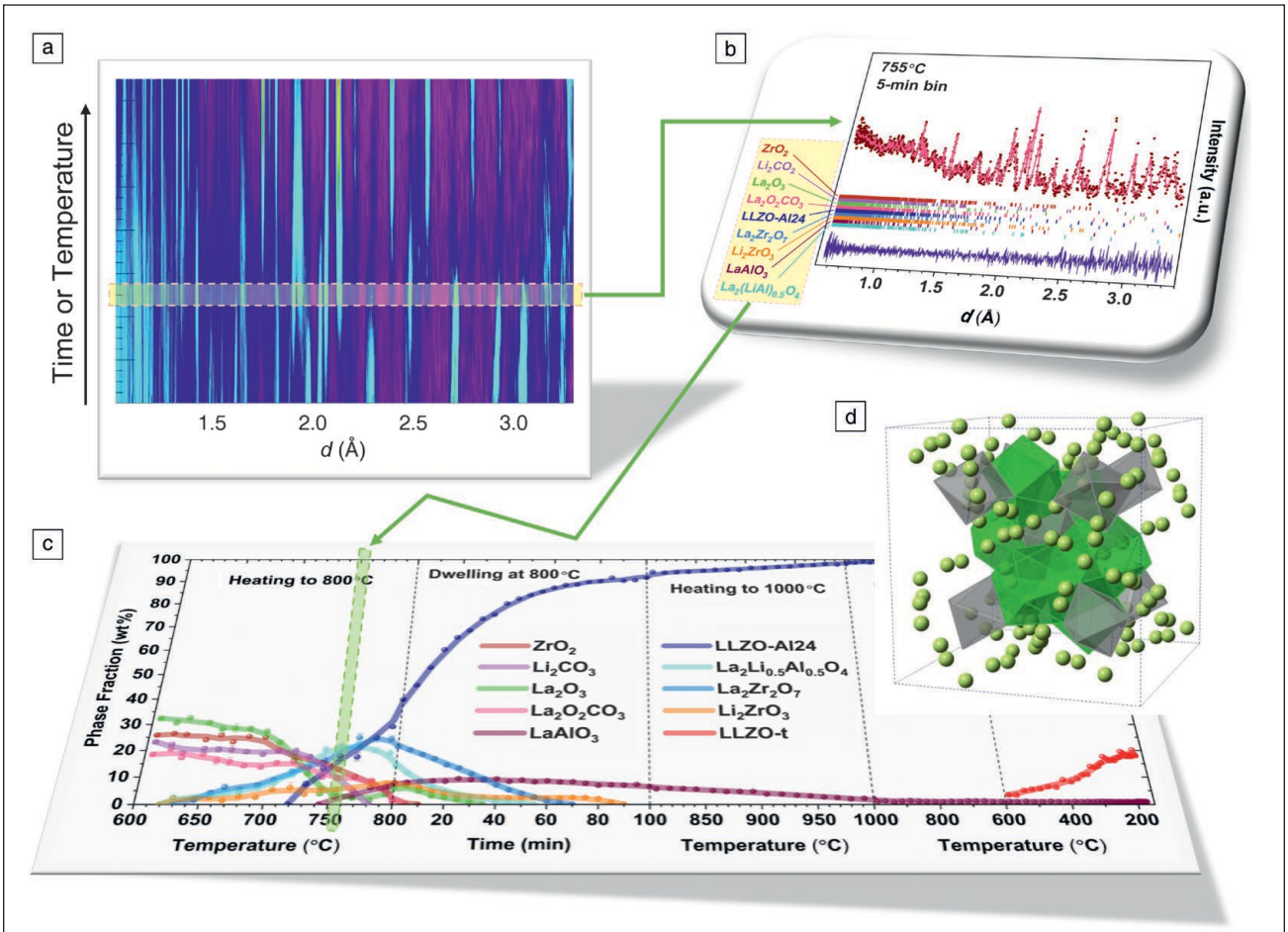


Figure 2. An *in situ* neutron work flow is demonstrated for the solid-state synthesis of Al-doped $Li_7La_3Zr_2O_{12}$ (LLZO-Al24) at high temperatures. (a) Neutron diffraction intensity contour plot versus lattice spacing (denoted by *d*) and time/temperature visualizes the phase evolution during synthesis. (b) An instant neutron diffraction pattern (5-min time bin) and Rietveld refinement identifies and quantifies the multiple phases at 755°C. (c) Refinement results of all instant neutron patterns reveal the mechanism and kinetics of LLZO-Al24 synthesis. (d) A schematic of the LLZO-Al24 garnet crystal structure shows the Li transport tunnels (green spheres indicating Li sites), as well as ZrO_6 polyhedra (gray) and LaO_8 polyhedra (green) in the framework.[39]

oxide is a high-voltage cathode material for Li-ion batteries in which the Ni and Mn arrangement influences the electrochemical performance.[13] The high contrast of Ni and Mn to neutrons enables quantification of the subtle ordering process when annealing at 700°C and a reversible structural transition to a rock-salt phase at higher temperatures.[12] The migration pathways of transition-metal ions among octahedral sites were found to use the tetrahedral vacancies as intermediate sites.[40]

The unique neutron capabilities of VULCAN have been exploited to understand functional material behaviors at high temperatures in a wide range of research areas, such as intermediate-temperature solid-oxide fuel-cell materials[41,42] and composites,[7] and thermoelectric materials.[43,44]

High-entropy alloys

High-entropy alloys (HEAs) are based on a novel alloy design concept—a solid solution with five or more principal elements that each has a large concentration of 5~35 at.%, thus possessing a high mixing entropy.[45] With a disordered global structure, an HEA possesses both high strength and high ductility at room temperature and elevated temperatures.[46] VULCAN can be used to reveal phase-specific and crystal-plane-specific lattice response[47] upon external heat and stress stimuli as a direct approach to investigate HEA formation, structure, physical properties, and performance over a wide temperature range. The structure–property relationship can be rapidly investigated. Plastic deformation mechanisms involving dislocations, stacking faults, twinning, phase transformation, and preferred grain orientation at different hardening stages can be elucidated by *in situ* neutron diffraction (e.g., by the transformation induced plasticity (TRIP) HEA in **Figure 3**).[28]

HTND was used to measure lattice parameter changes and the coefficients of the thermal expansion (CTE) of a family of Ni-containing single-phase solid-solution alloys with a change in entropy through the addition of more elements. The results confirm the estimation of the CTE by the quasi-harmonic approximation calculation with an error

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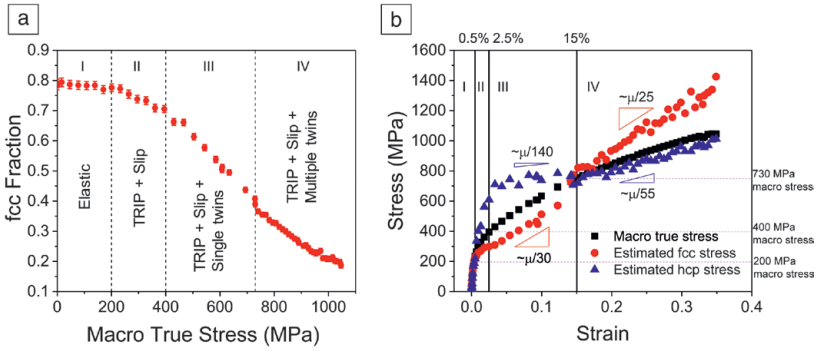


Figure 3. The staged deformation mechanisms and local phase stress and hardening revealed through neutron diffraction in a transformation induced plasticity (TRIP) high-entropy alloy. (a) The staged dominant deformation mechanisms are revealed in a TRIP high-entropy alloy. (b) The phase-specific stress–strain behaviors of each phase. The stress of face-centered-cubic (fcc) and hexagonal close-packed (hcp) phases can be calculated by multiplying the lattice strain (change in lattice parameter) and diffraction elastic constant. The phase-specific work-hardening rates μ are determined by the curve slopes and the triangles. The macro stresses and strains at the onsets of the stages are indicated. The results of these phase-specific deformation behaviors show that continuous fcc–hcp transformation and hcp phase work hardening contribute to the bulk work hardening.[28]

less than ~0.2%,[48] except for the case of Ni-20Cr, which was slightly underestimated by ~1%. The family of Ni-containing solid solutions exhibited similar CTEs, with the exception of strong magnetostriction effects for NiFe and NiCoFe in their ferromagnetic state; NiCoFeCrMn shows slightly higher CTE compared to others in this work.

During deformation, dominant plastic deformation mechanisms transition from one to the other, which is affected by both the temperature and straining ranges. Under uniaxial tension of NiCoFeCrMn HEA strengthened by carbon addition,[49] HTND showed the early stages of plastic deformation, where dislocation slip dominated the deformation behavior with little temperature dependency. However, under larger plastic strain, the deformation was governed solely by microbands at an elevated temperature while it was mediated simultaneously by deformation twins and microbands at room temperature.

HEA refractory alloys show promising high strength and ductility.[46,50–52] Micromechanics neutron measurements by measuring the change of crystal-plane-specific or *hkl*-specific lattice strains (*h*, *k*, and *l* are Miller indices denoting the crystal plane) during deformation showed that dislocation slip dominated the plastic deformation in a homogenized refractory NbTaTiV HEA,[46] and *in situ* neutron diffraction presented clear evidence of body-centered-cubic (bcc) to hexagonal close-packed (hcp) transformation and hcp and bcc micromechanical lattice strains behavior in the brittle-to-ductile deformation transition in tailored bcc TaHfZrTi HEAs.[50]

Superalloys

Superalloys including Ni-based, Fe-based, and Co-based alloys in single-crystalline, polycrystalline, or composite form are important high-temperature structural materials. They are widely used in aerospace, nuclear power generation, chemical, and petroleum industries. The understanding of their high-temperature behaviors is vital for energy efficiency, reliability, and safety. Neutron measurements of the bulk materials reveal changes in elastic constant and anisotropy,[53] misfit,[54] rafting,[55] phase stress sharing,[56–58] strengthening, and creep deformation mechanisms[59] over short- or long-term mechanical testing at both ambient and elevated temperatures, and development[58] and relaxation of residual stress[60] in thermomechanical treatments. Experimental micromechanical data from crystallographic measurements provide basic knowledge of high-temperature structural material behavior, accelerate alloy design, and yield direct experimental verification of predictive models of material and structure performance.

Additive manufacturing

AM or 3D printing of metals enables layer-by-layer fabrication of complex structures[61] using aluminum, stainless steel, titanium, and superalloys. The local rapid thermal and mechanical processes induce buildup of residual stress, distortion, metastable phases, and heterogeneous microstructure and texture, which inevitably impact the structural mechanical integrity in applications. Fundamental physical properties and engineering parameters, which govern the mechanical performance, need to be evaluated nondestructively. VULCAN probes the development of residual stress for essential computational model validation,[62,63] bulk texture measurements[64] for the development of elastic anisotropy,[65] hardening[66] and deformation in printed 3D structures,[67] and abnormal high-temperature deformation behaviors, as compared to traditionally manufactured alloys.[68]

VULCAN has seen continuous increases in demand for characterizing AM samples and structures fabricated from different alloys and their changes during and after heat treatments. The high-flux neutrons and instrument layout enable rapid lattice strain mapping deep inside structures with small voxels and simultaneously reveal heterogeneous distribution of the local phase and grain orientation texture in additively manufactured structures. Rapid time-dependent and nonequilibrium phenomena such as diffusion or phase transition can be observed directly or stroboscopically by native time-event data collections on the instrument. In addition, by combining neutron diffraction with neutron imaging,[69–71] time-of-flight neutron instruments, such as VULCAN, open unique scientific opportunities for high spatial resolution structural analysis in AM parts.

Outlook

The SNS at ORNL plans to upgrade the accelerator power from its current 1.4 MW to 2.8 MW to support a future second target station.[72] This will lead to 2.0-MW beam power

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on the first target station. VULCAN is currently upgrading to high-spatial-resolution and high-efficiency ^{3}He detectors with wider detector coverage. Both upgrades would increase the measurement rate by a minimum of a factor of two. The detector upgrade would also enable pinhole neutron diffraction to have high spatial strain measurement[73] down to 250 μm, as compared to the current value of 2 mm, and small-angle scattering for multiscale measurements. *Operando* materials response such as in a running engine, or during AM, will be possible to observe with these future instrument capabilities.

Acknowledgment

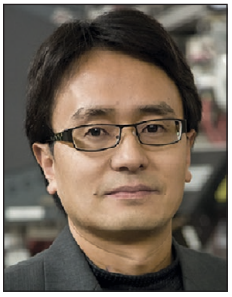
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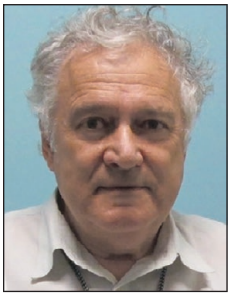
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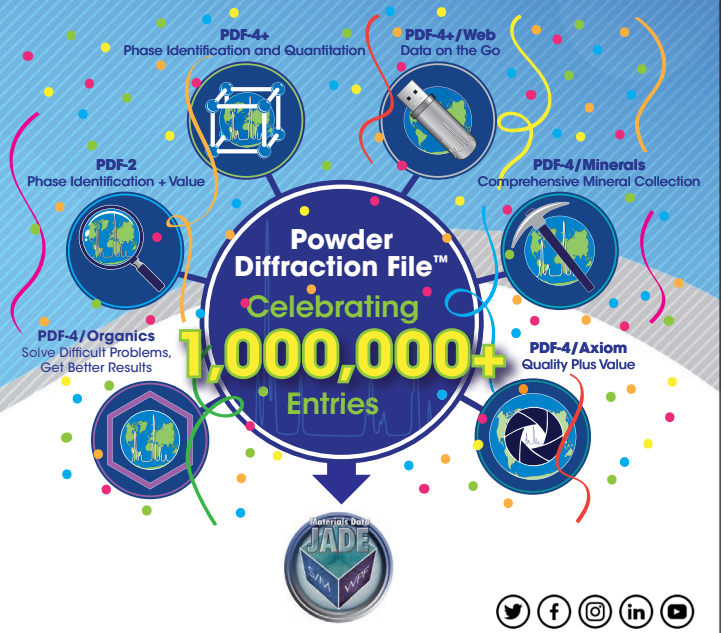
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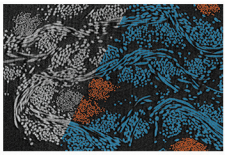
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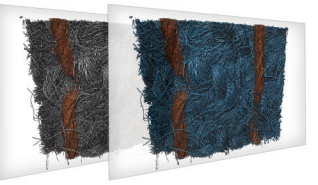
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