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Dynamic strength enhancement and strain rate sensitivity in ice-templated ceramics processed with and without anisometric particles

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A R T I C L E I N F O

ABSTRACT

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Keywords: Ice-templating Platelets Compressive response Strain rate Strain rate sensitivity We investigated the effects of platelets and strain rate on the compressive response of ice-templated porous alumina, processed from ultrafine, equiaxed particles and from a mixture of ultrafine, equiaxed and large platelet particles. Results revealed a remarkable enhancement of the quasistatic and dynamic compressive mechanical properties of the templated ceramics in the presence of platelets. Specific compressive mechanical properties of the ice-templated alumina containing platelets are superior in comparison to those of the various metallic, syntactic and natural cellular solids. In the presence of platelets, compressive strength exhibited contrasting strain rate sensitivity between the elastic and inelastic stages of deformation.

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absorption capacity (area under the stress-strain curve). To utilize

platelets-reinforced ice-templated hierarchical lightweight ceramic ma-

terials in the dynamic environments in which materials are subjected to

mechanical forces in tens of microseconds (µs), it is necessary to inves-

tigate compressive mechanical properties in the high-strain rate regime $(\geq 10^2 \text{ s}^{-1})$ that is drastically greater relative to that encountered under

quasistatic loading conditions, strain rate $\sim 10^{-3}$ s⁻¹. Due to the signifi-

cant strain rate difference, compressive deformation response of cellular

and porous solids measured in the quasistatic strain rate regime does

not truly represent the material behavior in the dynamic loading regime

[11, 12]. In a recent study [13], we showed that compressive mechanical

properties of ice-templated Al₂O₃ materials are considerably strain rate

sensitive. In this letter, we report on the dynamic strength enhance-

ment, progressive deformation characteristics, energy absorption, and

Significant interest in ice-templated macroporous ceramic materials has arisen primarily from their inherent hierarchical, anisotropic microstructure, directional porosity and low pore tortuosity (i.e., high pore connectivity), which are deemed suitable to enhance the performance of porous materials in biomedical, defense, energy, filtration, and other engineering applications [1–5]. In these engineering endeavors, compressive mechanical properties are of major significance to ensure the mechanical reliability of ice-templated macroporous ceramic components. Compressive response of ice-templated sintered macroporous ceramic materials features an approximately linear increase of stress with strain up to a maximum compressive stress (referred to as peak stress) and exhibits a graceful progressive failure beyond maximum compressive stress, as such further compression leads to a gradual decrease of stress with increasing strain [4, 6, 7]. At a specific level of porosity (i.e., material density) processing mechanisms capable of enhancing compressive mechanical properties of ice-templated ceramic materials are of great interest. Ice-templated ceramics are typically processed from aqueous suspensions containing fine, equiaxed ceramic particles [1–7]. In our recent studies [8–10], we revealed that inclusion of small fraction of large anisometric alumina (Al₂O₃) particles of platelet morphology in aqueous suspension that mainly consisted of fine, equiaxed Al₂O₃ particles led to two-fold unique microstructural effects in the templated sintered Al₂O₃ materials: substantial increase of lamellar bridge density and modification of lamella wall microstructure. The resulted sintered ceramic materials exhibited an unprecedented enhancement of uniaxial compressive strength, plateau stress, and energy

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strain rate sensitivity of ice-templated Al₂O₃ materials processed with and without the anisometric platelet particles. In this study, we synthesized ice-templated Al₂O₃ materials from aqueous suspensions containing (a) ultrafine, equiaxed Al₂O₃ (UA) powder particles ($d_{50} = 300$ nm) and (b) 80–20 vol% mixture of UA particles and large Al₂O₃ platelets (PA, diameter ~8 µm and thickness ~400 nm); however, total Al₂O₃ content in all the suspensions was same, 20 vol%. Ceramic suspensions were unidirectionally solidified at comparable freezing conditions and resulted average freezing-front velocity (FFV) was 38.9 ± 1.5 µm/s and 38.2 ± 1.5 µm/s for ice-templated UA materials and UA-PA materials, respectively. The templated materials were freeze-dried at 0.014 mbar pressure and -50 °C for 96 h and sintered at 1550 °C for 4 h. All the sintered materials were of cylindrical geometry (diameter ~10 mm and height ~18 mm). From each sintered material, a thin disk (2 mm) was extracted using a diamond









Fig. 1. Representative uniaxial compressive stress-strain curves of (a) UA material and UA-PA material in quasistatic regime of strain rate, (b) UA material both in quasistatic and dynamic regime of strain rate (~730–2200 s⁻¹), and (c) UA-PA material both in quasistatic and dynamic regime of strain rate (~7100–3200 s⁻¹). For both materials strain rate in quasistatic regime is ~0.002 s⁻¹.

saw and utilized for density measurement and uniaxial compression test. Quasistatic compression tests over a strain rate range of 10^{-4} – 10^{-1} s⁻¹ were conducted in mechanical testing machines (MTS Alliance RF/300 and Tinius Olsen 10ST), whereas a modified split-Hopkinson pressure bar (SHPB) was employed to perform dynamic compression experiments (strain rate $\geq 10^2$ s⁻¹). For both the dynamic and quasistatic tests, the compressive load was applied parallel to the growth direction of ice crystals. SHPB set up and working principles and other experimental details are provided in the Supplementary Material.

Average relative density (ρ_r) of ice-templated sintered UA material was found to be 0.3 (i.e., 70 vol% porosity), whereas ice-templated sintered UA-PA material exhibited an average density of 0.25 (i.e., 75 vol% porosity). While the average relative density difference is not drastic, results of density measurements suggest that presence of a small fraction of platelets increased porosity to an extent relative to the templated materials processed without platelets. Our earlier work on ice-templated Al₂O₃ materials [8–10] showed that large platelet particles formed lamellar bridges between adjacent ceramic lamella walls and significantly enhanced lamellar bridge density in comparison to Al₂O₃ materials processed without the platelets. Additionally, the presence of platelets increased porosity to an extent. In this study, we investigated the effects of anisometric platelet particles on both the uniaxial dynamic and quasistatic compressive response of ice-templated sintered Al₂O₃ materials.

Fig. 1a shows representative uniaxial quasistatic (strain rate ~0.002 s⁻¹) compressive response of ice-templated UA and UA-PA materials. For both UA and UA-PA, the compressive response shows approximately a linear increase of stress with strain and beyond peak stress (i.e., maximum compressive stress, $\sigma_{\rm p}$) a decrease of stress with increasing strain, followed by a densification stage in which stress increased sharply with increasing strain. At a strain rate level of 0.002 s⁻¹, UA-PA exhibited a drastic enhancement of $\sigma_{\rm p}$ relative to UA. As a result, the area under stress-strain curve up to the densification strain, which is a measure of the total energy absorption capacity, is also observed to be significantly greater for UA-PA relative to UA. Therefore, while relatively greater porosity of UA-PA in comparison to UA is expected to cause a decrease in strength for the former materials in comparison to the latter materials, the addition of small amount of anisometric PA particles resulted in a marked enhancement of compressive mechanical properties. While UA-PA exhibited a distinct strength advantage over UA, the addition of anisometric particles also resulted in an observable difference in the overall signature of compressive response in between the two materials. Beyond peak stress, further uniaxial compression resulted in a gradual decrease of stress with increasing strain for UA, whereas UA-PA exhibited a drastic decrease of stress with increasing strain. However, at any strain level, compressive strength of UA-PA remained significantly greater in comparison to UA. The uniaxial compressive response of UA-PA (porosity 75 vol%) can be characterized as a brittle-like failure behavior as opposed to the graceful progressive



Fig. 2. Variation in (a) peak stress, (b) specific peak stress, and (c) mass based energy absorption capacity with strain rate for UA and UA-PA materials.

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