



Full length article

Granular shape memory ceramic packings



Hang Z. Yu ^{a,1}, Mostafa Hassani-Gangaraj ^a, Zehui Du ^b, Chee Lip Gan ^{b,c},
Christopher A. Schuh ^{a,*}

^a Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139, USA

^b Temasek Laboratories, Nanyang Technological University, 637553, Singapore

^c School of Materials Science and Engineering, Nanyang Technological University, 639798, Singapore

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ABSTRACT

Although bulk shape memory ceramics (SMCs) are brittle, in particulate form they exhibit large recoverable strains in both shape memory and superelastic modes. Here, we investigate the fundamentals of mechanically- and thermally-triggered martensitic transformation of granular SMC packings. Specifically, $(\text{ZrO}_2)_{1-x}-(\text{CeO}_2)_x$ is studied in three different composition regimes. In the shape memory regime (below the martensite finish temperature), confined uniaxial compression leads to martensite re-orientation in the granular SMC packing, with the peak intensity of preferred crystallographic orientation increasing with external loading. In the intermediate regime (between austenite start and martensite start temperatures), confined uniaxial compression leads to irreversible martensitic transformation with the transformed volume increasing with external loading. This provides direct evidence of stress-induced martensitic transformation in granular SMCs. In the superelastic regime (above the austenite finish temperature), confined uniaxial compression leads to forward (during loading) and reverse (during unloading) martensitic transformation, manifesting in a large hysteresis loop in each load-unload cycle with remarkably high energy dissipation density. Based on finite element modeling of SMC particles in contact, we explore the martensitic transformation under non-uniform Hertzian stresses, which in turn provides insight on the experimental results.

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1. Introduction

Shape memory alloys exhibit two unique properties, namely the shape memory and superelastic effects, owing to a reversible martensitic transformation between two different crystallographic phases [1–10]. The forward martensitic transformation is characterized by martensite start and finish temperatures M_s and M_f , and the reverse transformation by austenite start and finish temperatures A_s and A_f . In the shape memory regime ($T < M_f$), the material is in the martensite phase and likely exhibits a variety of differently-oriented martensite variants, or twins; external loading leads to deformation by reorientation of the martensite variants to those

that best accommodate the applied strain, with an associated shape change. Subsequent heating reverts the ‘detwinned’ martensite to austenite. Upon cooling, austenite transforms back to twinned martensite without a shape change due to self-accommodation [11]. In the superelastic regime ($T > A_f$), the alloys are originally in the austenite state, and external loading leads to martensitic transformation. Upon unloading, the martensite transforms back to austenite. The martensitic phase transformation results in shear strains at the level of the crystal lattice [2,12], which can be realized up to the macroscopic scale; a remarkably high recoverable strain (as high as ~10%) can be obtained in either the thermally- or stress-activated cases.

With the capability of reversibly transforming between tetragonal and monoclinic phases, zirconia-based ceramics [6,12–19] represent a unique family of shape memory materials. Compared to conventional shape memory metal alloys [20,21], zirconia-based shape memory ceramics (SMCs) have a much higher transformation stress, a much larger hysteresis in a load-unload cycle, and a much wider range of tunable transformation temperatures, from room temperature to 1200 °C [14,15,22,23]. These properties

* Corresponding author.

E-mail address: schuh@mit.edu (C.A. Schuh).

¹ Current address: Department of Materials Science and Engineering, Virginia Tech, Blacksburg, VA 24061, USA.

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render SMCs ideal for a wide variety of emerging applications that are unaddressable by shape memory metals, such as ultra-high energy dissipation and high-temperature actuation.

As intrinsically brittle ceramics, SMCs are not likely to survive the transformation intact if they are large and polycrystalline, because the transformation strain is far beyond the elastic limit of the lattice. This is a key challenge with these materials. Practical SMCs are therefore most likely to involve small specimen length scales and few grains [6,24,25], so that when the transformation happens it is unconstrained and the transformation mismatch is accommodated at free surfaces. Lai et al. [6] first showed that in small zirconia micropillars with very few grains (i.e., oligocrystals or single crystals), the mismatch stresses associated with the transformation could be relieved at the pillar surfaces, permitting recoverable strains as high as ~7–8%. In superelastic cycling experiments, they demonstrated energy dissipation on the order of 100 MJ/m^3 , two to three orders of magnitude higher than that seen in shape memory metal alloys.

With the requirement of fine specimen size scales and few grains, approaches to scaling up SMCs for bulk applications are limited. One clear approach is to use SMCs in a granular form where every individual granular particle is a fine, oligocrystalline “specimen” of SMC. An aggregation of such granules could present an effective approach to bulk-level applications, such as high-energy dissipation and vibration damping. For example, high-energy dissipation armor may comprise an encapsulated granular SMC packing, or vibration-damping devices may incorporate such a packing between more rigid vibrating surfaces [26–29]. In these cases, the input mechanical energy would be converted to heat not only through friction amongst the particles as in conventional granular packings, but also through reversible, stress-induced martensitic transformation in individual particles when the critical transformation stress state is reached, as recently seen in cyclic compression of individual micro-scale superelastic particles [30]. This application would also inherently mitigate concerns about fracture; in contrast to the sensing and actuation applications typically targeted for shape memory materials [31–36], where fracture is detrimental, fracture could be beneficial for granular SMC-based energy dissipation devices, because fracture itself releases strain energy [37,38]. Fracture indeed would only serve to reduce the SMC particles' size, which in principle should enhance their superelastic properties on subsequent cycles.

The goal of this paper is to present the first systematic exploration of the mechanics and phase transformation behavior of granular SMCs under confined loading. Specifically, we study the mechanical and thermal responses of granular $(\text{ZrO}_2)_{1-x}(\text{CeO}_2)_x$ in three different compositions designed to access three distinct behavioral regimes (see Fig. 1(a)) including the conventional shape memory and superelastic regimes, as well as an intermediate regime that provides direct quantitative evidence for stress-induced martensitic transformation in SMC packings.

2. Experimental procedures

The $(\text{ZrO}_2)_{1-x}(\text{CeO}_2)_x$ -based SMCs are synthesized by chemical co-precipitation from two dissolved salts, $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$, with ammonium hydroxide, followed by washing, filtration, drying, calcination, and crystallization at $1500 \text{ }^\circ\text{C}$. The ceria atomic concentration x was controlled through the weight ratio of the two precursor salts as in Refs. [6,39]. By tuning the atomic concentration of ceria, we are able to investigate thermal and mechanical responses of SMCs in different regimes:

- *The shape memory regime* ($T < M_f$). In this regime, the SMC is stable in the monoclinic phase (i.e. martensite); external loading

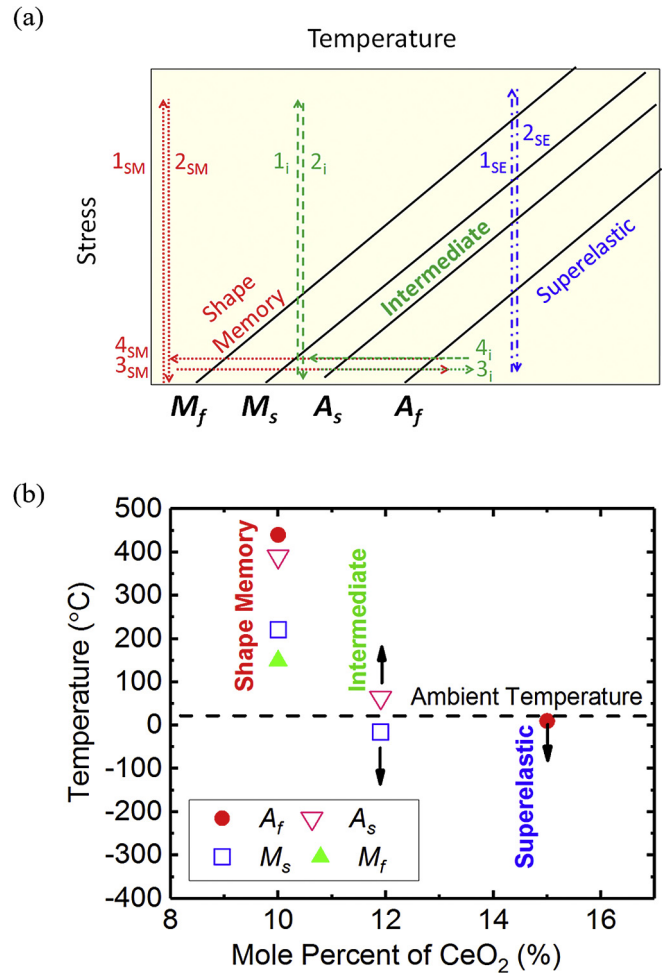


Fig. 1. (a) Stress-temperature diagram for SMCs. Arrows in different colors are used to illustrate experimental steps in the shape memory, intermediate, and superelastic regimes. (b) Transformation temperatures plotted for each of the shape memory, intermediate, and superelastic regimes investigated in this work.

may result in detwinning and martensite reorientation, but not phase transformation. For powders with $x = 10\%$ in $(\text{ZrO}_2)_{1-x}(\text{CeO}_2)_x$, the transformation temperatures have been measured using differential scanning calorimetry (DSC), with the martensite finish temperature $M_f \sim 150 \text{ }^\circ\text{C}$, martensite start temperature $M_s \sim 210 \text{ }^\circ\text{C}$, austenite start temperature $A_s \sim 380 \text{ }^\circ\text{C}$, and austenite finish temperature $A_f \sim 430 \text{ }^\circ\text{C}$. Since the martensite finish temperature is above room temperature, this composition produces a material in the shape memory regime at room temperature.

- *The intermediate regime* ($M_s < T < A_s$). In this regime, the SMC is metastable in the tetragonal phase (i.e. austenite). It transforms to the monoclinic phase when the applied stress reaches a critical value, but will not transform back to the tetragonal phase as the applied stress is removed. For powders with $x = 12\%$ in $(\text{ZrO}_2)_{1-x}(\text{CeO}_2)_x$, the as-prepared powders are already in the tetragonal phase from X-ray diffraction analysis. No martensitic transformation is detected upon cooling to $-170 \text{ }^\circ\text{C}$ in DSC, which may be caused by kinetic constraints for martensite nucleation at low temperatures. However, from X-ray diffraction analysis, we confirm that stress causes martensitic transformation in the $(\text{ZrO}_2)_{0.88}(\text{CeO}_2)_{0.12}$ powders at room temperature, which remain in the martensite state as the stress is removed. These will be shown in detail in Section 4. Therefore,

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