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## Failure mechanisms in thin-walled nanocrystalline cylinders under uniaxial compression

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Abstract—Lattice materials composed of hollow nanocrystalline struts have recently made it possible to access new regions of material property space, by exploiting structural efficiencies along multiple length scales (nanometre to centimetre range). An important design issue for these materials is to understand how the failure mechanisms that act at these scales affect the macroscopic mechanical properties. In this study, we tested hollow nanocrystalline cylinders of two different grain sizes (20 nm and 100 nm) in uniaxial compression to investigate the effect of grain size on dominant failure mechanisms, and the influence of the latter on the compressive strength. The finite element method was used to model the interaction of the three observed failure mechanisms: shell buckling  $(SB)$ , yielding  $(Y)$  and fracture  $(F)$ . Depending on the grain size and geometry, the failure sequence can be SB–Y–F, Y–SB–F, SB–Y or Y–SB, the order of which has important implications in defining the limits of mechanical performance. One such implication is that when shell buckling occurs in the inelastic regime of the material, the macroscopic strength increase due to grain size refinement can be greater than the inherent yield strength increase of the material. Second, material fracture and shell buckling may not be competing failure mechanisms, which means that the effectiveness of grain size reduction in increasing the structural efficiency of cylindrical strut members can span the entire Hall–Petch range of the material.

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### 1. Introduction

The failure of engineering materials occurs through processes that span a range of length scales. A central issue in materials design is the understanding of mechanisms by which processes that occur at one length scale influence those that act at others. Phenomena that bridge dimensional ranges are especially important in the development of internally architectured materials, which have structural features that are deliberately controlled to span orders of magnitude [\[1,2\].](#page--1-0)

One example of such materials is the class of microtruss assemblies, i.e. networks of beams, tubes or struts with characteristic dimensions in the nanometre to centimetre range [\[3–10\].](#page--1-0) Design efforts in this class of materials often involve three length scales. On the macroscopic (e.g. cm) scale, the architecture of the network is designed to be stretch-dominated so that externally applied loads are resolved axially along the interconnected network of internal struts; this strategy results in increasing the weight-specific strength and stiffness relative to cellular materials with bendingdominated deformation (see Refs. [\[1,2,11,12\]](#page--1-0) for examples).

On an intermediate (e.g. micrometre to centimetre) scale, a particularly effective design approach is to use strut members with functionally efficient cross-sections (e.g. hollow tubes). Design of cross-sectional efficiency at this intermediate scale is very effective in increasing the weight-specific properties of the structural members, because the axial buckling stress is proportional to the strut's radius of gyration (defined as  $\sqrt{I/A}$ , where I and A are the cross-sectional second moment of area and area, respectively), which means that a given mass of material would provide a substantially higher buckling resistance when positioned away from the neutral bending axis of the strut [\[13–17\].](#page--1-0)

A third level of design is directed at the scale of the grain size of the constituent material (e.g. nanometre to micrometre scale). One example is the deposition of high-strength, grain-refined sleeves on architecturally optimized precursor geometries, effectively creating an interconnected network of nanocrystalline tubes [\[5–10\].](#page--1-0) Using this concept, layers of Ni-based alloys with grain sizes in the 10-20 nm range have been deposited both on conventional foams  $[5,18]$ and microtruss architectures [\[6,19\],](#page--1-0) creating metal/metal and metal/polymer hybrids. This reinforcement is particularly useful because of the high yield strength achievable from grain size reduction to the nanometre scale, and the positioning of the reinforcement away from the neutral bending axis of the struts. Removal of the precursor after electrodeposition makes possible the fabrication of ultralight structures; recently, for example, microlattices with

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hollow tube morphologies were fabricated at densities as low as  $10^{-4}$  g cm<sup>-3</sup> [7-10].

The reduction of the material's grain size to the nanocrystalline regime introduces new failure mechanisms whose interaction and influence on mechanical properties are not well understood. Under uniaxial compression alone, examples of failure mechanisms include fracture at the strut connective regions  $[5-9]$ , local  $[20]$  or global  $[6,19]$  strut buckling and catastrophic fracture in the cylindrical walls [\[10,20\]](#page--1-0). In studies conducted so far, the role of the material's grain size on the failure mechanisms and mechanical properties of the cellular assembly has not been investigated. This analysis is complex due to two issues: the non-monotonic relationship between the strength of the assembly and that of the constitutive material, and the interaction between material failure and structural collapse.

First, the structural strength of stretching-dominated lattices is by definition determined by the axial deformation characteristics of the constituent struts, thus it is expected to increase in some proportion to the material's inherent yield strength increase owing to grain size refinement. However, in cases when instabilities occur in the inelastic regime of the material, the critical strength and strain of the column are dependent on both the geometry of the column (the slenderness ratio) and stress–strain relationship in the inelastic regime; the latter determines the function of the material's tangent modulus with strain. Generally, the work hardening capacity of the material is determined by the intracrystalline dislocation interaction, so a reduction in grain size also means that a lower tangent modulus is achieved.

Second, since the strength increase with grain size refinement is accompanied by a reduction in ductility, the underlying failure mechanism may change to fracture, e.g. resulting in progressive crushing [\[20\],](#page--1-0) catastrophic failure [\[20\]](#page--1-0) or localized end fracture [\[7,8\]](#page--1-0) of the hollow cylinders. However, the nature of the interaction between the two failure mechanisms is unclear, and so is their effect on the macroscopic strength of the cylinder. This is partially due to the unclear scaling of ductility with grain size. While the failure strain of nanocrystalline materials is typically lower than that of their polycrystalline counterparts  $[21–26]$ , there exists a plateau in the nanocrystalline regime (10-80 nm) [\[25,26\]](#page--1-0) where the intrinsic ductility ceases to be sensitive to grain size and becomes instead a strain-controlled phenomenon.

The mechanisms that control the compressive deformation of thin-walled nanocrystalline tubes are therefore influenced by properties in the microstructural and macroscopic length scales. This study provides a first examination of the effect of grain size on the inherent failure mechanisms and strength of these cylinders. Uniaxial compressive tests of specimens with two typical grain sizes (20 nm and 100 nm) are used to report the achieved strength and discuss its dependence on geometric and material properties. Finite element calculations are also conducted to gain insights into the interaction between macroscopic (structural) and microscopic (material) failure, with fracture described through a void growth model.

#### 2. Methods

#### 2.1. Experimental

Polymer substrate cylinders with radius of 3.5 mm were metallized and electroplated to varying thicknesses using a pulsed-current electrodeposition process; details of the procedure can be found in Refs. [\[27,28\].](#page--1-0) After electrodeposition the substrates were mechanically removed, resulting in standalone Ni hollow cylinders of inner radius 3.5 mm and four sets of wall thicknesses in the range of  $42 \pm 1$  µm to  $382 \pm 3$  µm, shown in Table 1 (for simplicity, the thicknesses are hereafter referred to as 40, 80, 190 and 380 µm). Two sample sets were fabricated, one having an average grain size of  $19 \pm 7$  nm (hereafter referred to as 20 nm Ni) and the other having a grain size of  $108 \pm 35$  nm (henceforth referred to as 100 nm Ni); these grain sizes are typical of Ni electroplated with these conditions [\[29,30\]](#page--1-0). The Ni tubes were then sectioned into specimens of 20 mm length and subsequently tested in uniaxial compression at a constant strain rate of  $10^{-3}$  s<sup>-1</sup>. To get statistically significant observations, at least five specimens for each grain size/thickness combination were tested.

For mechanical and microstructural characterization, flat sheets of free-standing Ni were also deposited using the same conditions. The microstructure was characterized by transmission electron microscopy (TEM) with electron transparent foils produced by twin-jet electropolishing. Bright-field TEM images of the two microstructures are presented in [Fig. 1](#page--1-0). The tensile properties of the two materials were obtained by testing coupons of a standard geometry at a strain rate of  $5 \times 10^{-4}$  s<sup>-1</sup> (see Ref. [\[26\]](#page--1-0) for details); the reference tensile curves are shown in [Fig. 2.](#page--1-0) The 20 nm grain size material had a 0.2% offset yield strength of 872 MPa and an elongation to failure of 0.06, whereas the 100 nm grain size material possessed a yield strength of 490 MPa and an elongation to failure of 0.16 [\[26\].](#page--1-0)

**Table 1.** Summary of grain size, cylinder thickness (*t*), peak nominal strength ( $\sigma_N^{PK}$ ), and failure mechanisms observed in the experimental tests and FE calculations of this study.

		Experimental		FE calculations	
Grain Size	$t \text{ } (\mu \text{m})$	$\sigma_N^{PK}$ (MPa)	Failure mechanisms	$\sigma_N^{PK}$ (MPa)	Failure mechanisms
$20 \text{ nm}$	$42 \pm 1$	$524 + 216$	4 fold	957	4 fold
	$81 \pm 1$	$847 + 147$	4 fold	1137	4 fold/failure
	$191 \pm 7$	$1323 + 177$	fracture	1377	2 fold/axi./failure
	$382 \pm 3$	$1294 + 93$	fracture	1445	axi./failure
$100 \text{ nm}$	$44 + 2$	$346 + 99$	4 fold	507	4 fold
	$86 \pm 1$	$502 + 50$	4 fold/3 fold	560	4 fold/3 fold
	$176 + 2$	$526 + 26$	3 fold/axi.	601	3 fold/axi.
	$372 \pm 3$	$654 \pm 21$	$2$ fold/axi.	686	2 fold/axi.

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