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# Mechanical properties of the ice I–magnesium sulfate eutectic: A comparison with freshwater ice in reference to Europa

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## ABSTRACT

A systematic series of experiments on laboratory grown ice I–magnesium sulfate (MgSO<sub>4</sub>·11H<sub>2</sub>O or MS11) eutectic columnar material and freshwater columnar ice at T = 98 K and T = 263 K directly compare some elastic and inelastic properties of these two materials. The results indicate: (i) that the ice I–MS11 eutectic columnar material (below simply as I–MS11) exhibits bulk elastic properties that are comparable to those of freshwater columnar ice (FWI) at T = 263 K; (ii) that I–MS11 is about 1.6 times stronger under bending, about 1.9 times stronger under compression within the brittle regime and about 1.3 times tougher than FWI at T = 263 K; (iii) that I–MS11 possesses a compressive flow strength within the ductile regime that is about one-half that of FWI at T = 263 K; (iv) that I–MS11 exhibits a transition from ductile-to-brittle behavior at a strain rate that is a factor of about 50 higher than that of FWI at T = 263 K; (v) that I–MS11 has a higher coefficient of kinetic friction at lower temperatures (T = 98 K) and a lower coefficient of kinetic friction at higher results indicate that the mechanical properties of I–MS11 eutectic columnar material are generally comparable to those of freshwater columnar ice, under the conditions examined.

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# 1. Introduction

As knowledge of the tectonic evolution and composition of icy satellites in the outer Solar System increases, there is an increasing need to determine the mechanical properties of the materials from which these satellites are composed. Europa, Jupiter's sixth moon, is a satellite of particular interest owing to the possibility of a subsurface ocean beneath its icy crust, as suggested by geological evidence (Greenberg et al., 1998; Pappalardo et al., 1999; Hoppa et al., 1999a,b; Sotin and Tobie, 2004) and magnetic evidence (Kivelson et al., 2000), and to the subsequent potential for underwater life (e.g. Figueredo et al., 2003; Tyler, 2008). Imaging from Galileo's Near-Infrared Mapping Spectrometer indicates asymmetric water absorption, which suggest that Europa's icy surface is composed of more than pure ice. Laboratory studies show that these absorption bands may be caused by the presence of hydrated sulfates, particularly of magnesium and sodium (Carlson et al., 2002). The presence of magnesium and sodium sulfate hydrates in the shell is also consistent with models of Europa's evolution (Kargel et al., 2000) and is plausible from a thermodynamic standpoint (Zolotov and Shock, 2001). Although no single compound matches

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the observed spectra perfectly, those with the highest level of hydration, such as  $MgSO_4 \cdot 11H_2O$  (termed meridianiite or MS11 by Peterson et al. (2007)), provide the best fit (Dalton et al., 2005). Given the spectra and assuming the presence of some liquid water, it is possible that regions of two-phase ice I–MS11 may exist and could have developed via eutectic solidification. Should Europa's crust have formed through unidirectional solidification, similar to the near-surface water freezing of Earth's oceans in the polar regions, the material could have developed a columnar structure.

Despite the evidence that Europa's crust is composed of more than single-phase water ice, current modeling of its tectonic evolution rests principally on the measured properties of "pure" freshwater ice. For example, quantitative analyses on the formation of Europa's double ridges, "the most ubiquitous landform on Europa" (Dombard et al., 2013), rely on material properties, including Young's modulus, the coefficient of friction, fracture toughness, tensile strength and the ductile-to-brittle transition strain rate, that have been determined for freshwater ice (see Dombard et al. (2013) for a recent review of models of double ridge formation). This approach is of course reasonable, given the paucity of information on the behavior of multi-phase ice. Yet, as others before us have noted (e.g., McCarthy et al., 2007, 2011), the presence of a second phase can affect material properties, often significantly. For instance, wood derivatives such as pulp and sawdust at volume

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fractions up to 0.1–0.2 raise the fracture toughness of water ice by about an order of magnitude (Nixon and Smith, 1987). Should an effect of that kind and magnitude translate to Europa and to its putative ocean, it could lead to a prediction of greater absorption by the icy crust of tidal-induced energy and correspondingly to an expectation of a larger ocean (Tobie et al., 2003; McCarthy et al., 2007). The question, then, is: just how different are the mechanical properties of a multi-phase ice, particularly a mixture of water ice and hydrated sulfates, from those of "pure" ice?

It is with this question in mind that the present work was undertaken. Specifically, the objective was to measure a range of mechanical properties of an eutectic composite composed of columnar-grained ice-I and MS11 (termed below simply as I– MS11), as prepared through unidirectional solidification, and to compare the properties to those of single-phase ice-I using the same procedures and under the same conditions. We report our results in this paper. Included for the first time are measurements of the elastic properties of I–MS11 as well as measurements of bend and compressive strength, fracture toughness, the coefficient of kinetic friction and the ductile-to-brittle transition.

#### 2. Experimental procedure

# 2.1. The ice

Columnar-grained material of the binary ice I-MS11 system (equilibrium eutectic temperature  $T_{eutectic}$  = 269.6 K) was grown in the laboratory through unidirectional solidification of a homogeneous liquid of eutectic composition, as follows: Approximately 2 L of de-ionized water was thoroughly mixed with 17.5 wt% of 99% pure anhydrous MgSO<sub>4</sub> powder (obtained from Acros Organics) at room temperature. The solution was poured into a cylindrical mold, containing a rough aluminum bottom and wellinsulated sides and lid. The bottom of the mold was affixed to a cold plate maintained at  $T = 253 \pm 0.1$  K. using a NESLab RTE-140 unit to circulate a chilled glycol-water solution. The growth apparatus was situated in a walk-in cold room built by Norlake Scientific Refrigeration Equipment and air temperature was maintained at  $T = 263 \pm 0.2$  K, as verified by an independent series of calibrated thermistors (sensitive to ±0.05 K). Freezing from the bottom up was allowed to continue for 48 h, after which the solidified eutectic material (a puck of 150 mm diameter and 70 mm thick) was removed. Thin sections confirmed a uniform columnar structure, with the exception of a ~5 mm layer of irregular structure (Fig. 1b) on the bottom and top.<sup>1</sup> The diameter of the columnar-shaped regions, as measured in a plane normal to the direction of growth using the linear intercept method, increased from  $d = 1.0 \pm 0.2$  mm near the bottom of the puck to  $d = 2.1 \pm 0.2$  mm near the top. The density of the material was  $\rho$  = 1048.8 ± 6.8 kg m<sup>-3</sup> at *T* = 263 K, as determined by weight and dimensions.

Columnar-grained freshwater ice was grown in the laboratory through unidirectional solidification, using established techniques described elsewhere (Golding et al., 2010). The ice was transparent and free from cracks, with a density of  $\rho = 914.1 \pm 1.6 \text{ kg m}^{-3}$  at T = 263 K. Column diameter increased from  $d = 3 \pm 0.5 \text{ mm}$  at the bottom of the specimens (described below) to  $d = 6 \pm 0.5 \text{ mm}$  near the top. Microstructural analysis using a Rigsby stage confirmed that the ice possessed the expected S2 growth texture, in which the crystallographic *c*-axes were confined to the horizontal plane of the ice, but randomly oriented within that plane.



**Fig. 1.** Thin section photographs of I–MS11 eutectic columnar material as viewed (a) on a section taken perpendicular to and (b) on a section taken parallel to the direction of solidification.

## 2.2. Microscopy and imaging

The microstructure of the I–MS11 eutectic material was examined using thin sections ( $\approx$  1 mm thick), as prepared with a Leitz microtome in a cold room maintained at *T* = 263 ± 0.5 K. High magnification images were obtained using a scanning electron microscope (SEM) equipped with a Gatan-C1001 cryogenic stage at *T* = 153 ± 4 K. Energy dispersive spectroscopy (EDS) was performed to determine phase composition. Image analysis and area fractions were computed using the program ImageJ.

# 2.3. Mechanical testing

All stages of specimen preparation and mechanical testing were performed in one of several Norlake walk-in cold rooms. Rectangular prismatic-shaped specimens were machined using a Hardinge horizontal mill at  $T = 263 \pm 0.5$  K. Specimen surfaces were maintained parallel within  $10^{-3}$  radians, as determined using a dial gage, with one surface cut perpendicular to the direction of column growth (i.e. perpendicular to the columnar microstructure). All tests were performed on both I–MS11 columnar material and freshwater S2 columnar ice.

#### 2.3.1. Elastic properties

Bulk elastic properties were measured dynamically through ultrasonic velocity measurements of compression and shear waves, by applying standard relationships (Timoshenko and Goodier, 1934), using an ULT-100 ultrasonic velocity test system manufactured by GCTS. The ultrasonic apparatus was calibrated using cylinders of copper and cylinders and rectangular prisms of aluminum. Due to a high level of acoustic noise, inherent in the two phase I–MS11 material, and the resulting uncertainties in the time of the returning peak, error in the elastic properties are estimated to be as high as 5% for the I–MS11 material. For freshwater ice, signal noise is estimated to be closer to 2% of the measured value. Ultrasonic measurements were made, both along and across the columns, at  $T = 263 \pm 0.2$  K using rectangular prismatic-shaped specimens of height whose dimension ( $h \approx 120$  mm) was maintained between two to three times greater than either dimension

<sup>&</sup>lt;sup>1</sup> Both irregular layers were removed from the material during specimen preparation (more below) and prior to mechanical testing.

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