Available online at www.sciencedirect.com

ScienceDirect

Acta Materialia 71 (2014) 185–194

Acta MATERIALIA

www.elsevier.com/locate/actamat

Hot-pressure sintering of low-density snow analyzed by X-ray microtomography and in situ microcompression

Stefan Schleef, Henning Löwe *, Martin Schneebeli

WSL Institute for Snow and Avalanche Research SLF, Davos, Switzerland

Received 29 November 2013; received in revised form 14 February 2014; accepted 2 March 2014
Available online 3 April 2014

Abstract

We analyzed the impact of confined compression at different strain rates on the microstructure of low-density snow. Experimental conditions in a cold laboratory with fragile snow samples at porosities of almost 0.9 required the construction of a new microcompression device for in situ microtomography measurements. Under isothermal conditions, compression experiments were conducted at low, constant velocities of 0–0.4 μm s^{-1} up to macroscopic stresses of about 13 kPa, followed by subsequent stress relaxation measurements. During compression, the temporal evolution of the stress for different strain rates follows a universal loading curve after rescaling. During relaxation, a different scaling is observed, which is quantitatively analyzed within a Maxwell model, indicating nonlinear viscoplastic behavior with $\dot{\varepsilon} \sim \sigma^n$ and a stress exponent of around $n \approx 2.2$. The main differences between compression and relaxation are revealed by the microstructural analysis of the tomography data. The specific surface area shows a rate-dependent decrease during compression. The decrease is related to the evolution of the topology of the ice matrix, suggesting a decrease in the surface area at the cost of grain boundary area creation due to the formation of new contacts. The significant structural changes are absent during the relaxation stage, when only minor topological coarsening effects remain and stresses can relax in an almost invariant microstructure.

Keywords: Coarsening; Compression test; Porous material; Ice; X-ray tomography

1. Introduction

Ice is gaining interest as a model material in material science for studying the mechanical behavior of polycrystalline solids under deformation at high homologous temperature [1–3]. Snow consist of ice grains connected in highly heterogeneous microstructures, constituting the high-porosity variant of this model material. Under isothermal conditions, snow crystals sinter and densify into a more compact porous structure. This process originates from the combination of stress-induced viscoplastic deformations and coarsening caused by the reduction of surface energy, and is commonly referred to as destructive snow metamorphism. As for any porous materials, the interplay between macroscopic stresses and strain rates on the one hand and the evolution of the microstructure on the other hand constitutes the main challenge for the material snow.

For snowpacks, there have been many studies on the mechanical behavior of snow [4–8] and its densification [9–11]. However, these studies focused on the macroscopic parameters and did not measure the evolution of the microstructure of snow. For the observation of the microstructure of solid materials, X-ray microtomography (μCT) has been established as a widely used method in many different fields of material science [12]. One advantage it has over other methods is that it is non-destructive, and therefore allows the evolution of samples to be observed under different conditions [13,14]. For snow, μCT is well suited for studying the continuing structural changes

* Corresponding author.
E-mail address: loewe@slf.ch (H. Löwe).

http://dx.doi.org/10.1016/j.actamat.2014.03.004
1359-6454/

[15–17] and analyzing the evolution of important parameters like density [18] and strain or the specific surface area (SSA) as an indicator for isothermal metamorphism [19–21]. A full 3-D reconstruction via μCT also allows the analysis of the topology of a structure [22], and has already been used for higher-density snow (firn) [23].

High-density snow subjected to high stresses has been investigated mainly in view of the transition from snow to firn and ice [24–27], and creep experiments for round-grained snow of intermediate density were conducted by [28]. With increasing porosity, however, in particular for new snow, the complex shapes of the original snow crystals dominate the structure and coarsening is of major importance. For new snow, the densification under low stresses was recently studied using creep experiments and μCT [29]. It was found that metamorphism is independent of the densification, and the decrease in the SSA is independent of the applied stress. In these experiments the stress was applied by dead loads on top of snow samples. The analysis was limited to low constant stresses, because large loads applied instantaneously cause the structure to collapse. Such instantaneous fracture processes have been studied from a macroscopic viewpoint [30] or, at high strain rates, by stereology for dense snow, in view of the microstructure [31]. To observe the gradual microstructural response during slowly increasing stress at low strain rates, it is, however, necessary to use displacement-controlled deformations.

Displacement-controlled experiments can be conducted with a microcompression device (MCD). The use of MCDs in combination with μCT is commonly applied in various fields of material science. The deformation of foams is studied with in situ compression using tomography beamlines at synchrotrons [32,33]. MCDs designed for use in commercial desktop devices are also used for foams and cellular solids [34]. However, these examples were performed with stepwise compression (ex situ). Further examples for in situ compression together with synchrotron as well as laboratory X-ray sources are given by [35]. The effects of stress on sintering were addressed by [36], who studied the in situ compression of polymer scaffolds with a synchrotron source by image-guided failure assessment. They observed sintering for low strain rates, in contrast to cracking at higher strain rates.

The observation of creep and sintering of the ice structure in snow in the absence of macrocracking requires low strain rates and low stresses. It was the aim of the present paper to analyze the effect of different strain rates during compression on the deformation and the sintering of low-density snow as an example of a highly porous polycrystalline material. To analyze the stress response, we built an MCD which enables in situ compression experiments of the tenuous snow structure in a desktop μCT device, which is operated in a cold laboratory under isothermal conditions for sample preparation and measurements. Our MCD allows us to compress snow samples at different low rates without collapse while measuring the resulting stress. Simultaneous series of μCT measurements provide a visualization of the structural changes and allow the calculation of the main structural parameters, such as SSA and topology, which are related to macroscopic stresses and strains.

2. Methods

2.1. Microcompression device

To measure low-density snow in a cold laboratory, we developed a microcompression device (MCD) tailored to the requirements of the material. The idea was to load the device in the cold laboratory carefully outside the tomograph in a rack without damaging the tenuous ice structure. A piston is then moved onto the snow surface to fix the ice structure, before mounting the MCD for the μCT measurements. The μCT scans were conducted with a desktop tomography device (μCT80, SCANCO Medical AG).

The MCD, shown in Fig. 1, is made of a cylindrical aluminum body with a maximum diameter of 120 mm, which allows free rotation in the μCT. On the top of the MCD (when mounted in the μCT) is a piston with a polyoxymethylene (POM) surface of 35.5 mm diameter that is driven onto the snow sample. The sample holder is made of 3.5 mm thick POM, which provides mechanical stability and undisturbed μCT scanning. A small gap between the diameter of the piston and the sample holder guarantees friction-free movement and air exchange. The maximum sample height is 30 mm, and the piston can be moved up to 11 mm.

The mechanical construction enables precise linear displacement of the piston with a DC step motor (maxon A-max, EPOS controller). An end switch defines the starting position of an experiment. Note that the mechanics

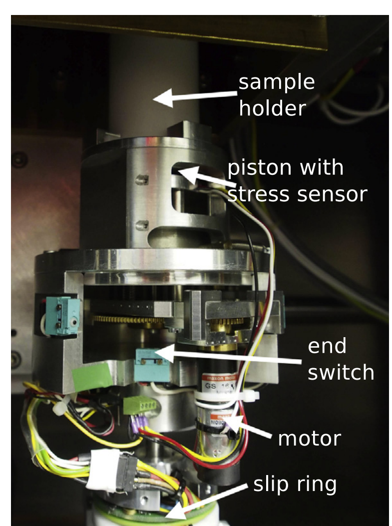

Fig. 1. Picture of the MCD operated in the μCT. The main components are indicated. The motor controller is not visible at the backside.