Research Article

Aerogels Materials as Space Debris Collectors

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Material degradation due to the specific space environment becomes a key parameter for space missions. The use of large surface of brittle materials on satellites can produce, if impacted by hypervelocity particles, ejected volumes of matter 100 times higher than the impacting one. The presented work is devoted to the use of silica aerogels as passive detectors. Aerogels have been exposed to the low earth orbit of the ISS for 18 months. The study describes the aerogels process and the choice of synthesis parameters in such a way to get expected features in terms of porosity, mechanical properties, internal stresses, and transparency. Low-density aerogels (0.09 g cm\(^{-3}\)) have been prepared. The control of transparency necessary to see and identify particles and fragments collected is obtained using a base catalysis during gel synthesis. After return to earth, the aerogels samples have been observed using optical microscopy to detect and quantify craters on the exposed surface. First results obtained on a small part of the aerogels indicate a large number of debris collected in the materials.

1. Introduction

Material exposure and degradation experiment (MEDET) is devoted to characterize the local environment of the International Space Station (ISS) and space environment especially in terms of debris and microparticles. Following satellites number increase and last explosions on orbit, the population is suspected to increase in particular \(\mu\)m size to mm particles. The risk is that these fragments are supposed to be able to damage the satellites surfaces (thermal protections, optics, and antennas) on orbit. The risk for extra vehicle activities (EVA) by astronauts is also taken in consideration. Figure 1 shows the effect of an impact of debris on a solar cell. Aerogel detectors are candidates for this mission. Because of their high compliance aerogels should be able to limit the shock during the impact. In previous works [1, 2] it has been shown that for hypervelocity cosmic particles with velocity close to 6 Km/s, intact recovery of particles is favoured with polystyrene foam having a bulk density close to 0.07 g cm\(^{-3}\).

Previous studies on aerogels [1, 3] have shown that for velocity in the range 5-6 km s\(^{-1}\), aerogels with bulk density higher than 0.06 g cm\(^{-3}\) have a better capture efficiency. Taking into account model prevision [4] of length track and intact recovery of particles, aerogels with density in the range 0.06–0.1 g cm\(^{-3}\) should be a good compromise. However, during aerogels synthesis internal stresses could appear because of a syneresis effect and associated shrinkage. The syneresis is the continuation of the hydrolysis and condensation reactions after gelling which leads to the gel shrinkage [5]. These internal stresses could be a risk for the integrity of aerogels in the case of a hard shock during the impact. Moreover important shrinkage will affect the control of the target density.

The present work will prepare low-density aerogel in the ranges 0.05–0.2 g cm\(^{-3}\) and characterize their mechanical properties (elastic modulus, strength). In this study is the influence of the chemical parameters (organosiloxane concentration and pH) on the shrinkage during the aerogels...
synthesis characterized. The control of aerogel transparency necessary to see and identify the particles and fragments will be done by the use of a base catalysis.

2. Experimental

The first step of the aerogel synthesis is the formation of a gel which involves the hydrolysis reaction of an organometallic compound (Si(OR)₄), dissolved in alcohol in the presence of water. By condensation reactions two silanol groups give rise to siloxane bonds [5]. In this work the gels are prepared as follows. Silicon tetraethoxide (TEOS) is hydrolyzed with water (4 moles of water per moles of TEOS) under stirring. The pH of the water is adjusted between 2 and 9 with different concentrations of nitric acid and ammonia. The TEOS concentration ranges between 10 and 46 volume %.

After gelling we obtain an alcogel (wet gel) which is a two-phase medium containing the solid network and the liquid (alcohol + water). A classical drying treatment can be carried out at ambient temperature but during this drying stage capillary stresses break the solid network [6, 7]. The supercritical drying (SD) eliminates these capillary forces because the pressure and the temperature pass over the critical point of the liquid. The aerogels are obtained after a SD performed by heating the gels to 300°C and 13 MPa. The heating rate was 1.5°C mn⁻¹, and the pressurization rate was close to 0.8 MPa mn⁻¹. Then the pressure is released by a controlled depressurization rate over a period of 13 hours. Details are presented elsewhere [8].

Since shrinkage was observed to be isotropic within experimental accuracy [9, 10], it was determined simply by measuring the length of the bar of gel (average data of 3 gel samples). The uncertainty in linear shrinkage is less than 5%. The bulk density is calculated from the weight and the dimensions of the samples (average data of 3 aerogel samples). The uncertainty in density is less than 3%. To characterize the mechanical properties, the elastic modulus E and the rupture strength (σ) were measured by a three point bending technique using an Instron testing machine [11]. Five experiments have been done on each sample, and the data dispersion is 20%. The transparency is characterized on aerogels slice of 1 mm thickness by the transmission in the UV-visible range (Beckman spectrometer) in the spectra range 0.2–0.8 microns.

3. Results

Taking into account the high velocity (5–10 km/s) and the mean size of debris (0.1 mm) which should be collected by the aerogels collectors, intact recovery of debris and particles is favoured in materials having a bulk density close to 0.07–0.1 g/cm³. An important point is the synthesis of aerogels with acceptable mechanical properties because the sensor should survive to 18 month in stressful conditions. It is easy to adjust the aerogel bulk density (pore volume) by the TEOS concentration [5–7]. Moreover, it is obvious that aerogel mechanical features are controlled by the aerogel bulk density [11, 12].

Our results (Figures 2 and 3) confirm that the bulk density, the elastic modulus, and the mechanical strength can be adjusted by the control of the TEOS content in the gelling solution.

However, in previous works it has been shown that some monolithic aerogels can crack several months later [8, 12] because of microflaws propagation. This behavior is the result of internal stresses and progressive rupture of siloxane bonds.
bonds. These internal stresses could be a serious drawback for aerogel sensors because they enhance the risk of aerogel destruction if the impacts induce flaw growth and the breaking up of the aerogel network. Generally internal stresses are the results of the syneresis effect [5] and the associated shrinkage occurring during the supercritical drying [8]. Thus, to have a precise control of the bulk density and favor the synthesis of aerogels without internal stresses, it is necessary to minimize this shrinkage.

Table 1 shows that the decrease of the TEOS concentration decreases the linear shrinkage. This shrinkage during supercritical drying can be explained if we consider that the heating treatments accelerate the condensation reactions. The structure of gels elaborated in neutral conditions is expected to consist of relatively flexible chains whose surface is covered by SiOH groups [5, 13], and shrinkage results from condensation between neighbouring groups. When two branches come into contact, condensation reactions of silanol groups take place increasing the connectivity. This process explains the large shrinkage and imposes internal stress on the gel network. The condensation rate increases with TEOS since the chains are closer together when the silica concentration is high. So, for high TEOS concentrations the number of condensation reactions and consequently the shrinkage are increased.

In basic conditions the shrinkage is lowered (Table 2). At high pH the network consists of larger particles, and the branches are stiffer and more widely spaced than in acidic or neutral conditions [5, 13–15]. The formation of larger silica particles hinders the shrinkage, because the larger silica particles and stiffer chains oppose to a restructuring. This mechanism explains the evolution of the shrinkage with the synthesis parameters. So, low silica concentration and basic catalysis limit the shrinkage, allow a better control of the sample volume, and decrease the internal stresses.

The last important feature for the aerogel collector is its transparency which will allow the easy localization of the debris inside the aerogel network. Let us consider the transmission properties of aerogels in the UV-visible range. Figure 4 shows the transmission spectra of a set of aerogels with different TEOS concentrations. The aerogels of 46% of TEOS (sample E) have a wider transmission window than the lower TEOS concentration sample (18% TEOS, sample A) which shows poor transparency in the visible range. These spectra report that the transmission window decreases with the TEOS concentration and the losses by scattering are higher for the lighter aerogels. In the literature it has been explained that the lightest aerogels contain a greater proportion of large pores (scatters) which make them less translucent [16].

A remarkable effect is observed when aerogels are prepared with ammonia catalysis, and Figure 5 reports that the addition of ammonia strongly improves the transmission
in the visible range. The aerogel becomes transparent and comparable to a glass. This is due to a low proportion of very large mesopores measured in basic aerogels compared to neutral ones [17].

These results show that chemical manipulation of starting solutions may substantially modify the characteristics of the aerogels collectors. So, in terms of bulk density, mechanical properties, internal stresses, and transparency the aerogel synthesized in basic conditions with a TEOS concentration equal to 18% is the suitable compromise.

4. Discussion

In the literature, aerogels are generally described as elastic and brittle materials like glasses, but the large porosity results in a poor “load-bearing fraction of solid” which leads to low elastic modulus and rupture strength [18–20]. Another parameter characteristic of the mechanical and brittle behavior of silica aerogels is the flaws propagation. Microlaws act as stress concentrators, and internal stresses favor the flaws growth up to the rupture. It is clear that these peculiar features: poor load-bearing fraction of solid and high susceptibility to flaw propagation, are important drawbacks to aerogels applications.

A bulk density close to 0.08 g·cm$^{-3}$ could be a good compromise between low bulk density (in such a way to allow the capture of debris) and acceptable mechanical properties. The present work thus defined the TEOS concentration (18%) allowing the synthesis of aerogels with the target density. Important syneresis effect occurs during the supercritical drying with a shrinkage as high as 30 linear percent. This process is accelerated by the high temperature and pressure [14].

Acid and neutral gels are described as made of small primary particles forming polymeric chains [13]. The flexibility of the chains allows contact and condensation reactions. For basic catalysis the solubility of silica is enhanced [14]; then the network is built by larger particles [13, 15], and the branched parts are more widely spaced limiting the possibility of new bonds formation. To minimize the syneresis shrinkage and the internal stresses the synthesis in basic conditions is required.

The transparency of the aerogels will depend on the absence of light scatters ( pores and/or aggregates) having a size in the range of 50–100 nm. In such a way to control the aerogels transparency, it is important to understand how the chemical conditions synthesis affects the aerogel structure ( pores and aggregates). Structural results by Small Angle X ray Scattering (SAXS) will help us to characterize the aerogel structure in the range of 1 nm–100 nm. SAXS experiments generally give information on the main features of the aerogel structure: the mean size of the aggregates which are connected to form the network and the mean size of the primary particles which stick together to build the aggregate. The aerogel network can be described as an assembly of aggregates (~10–100 nm) which are the result of the aggregation of small particles (1-2 nm). Scattering studies [21–23] have shown that the size of the aggregates increases when the organosiloxane concentration decreases, and for low concentration of neutral catalyzed aerogels of this study (10–18%) the mean size of the aggregates could be as high as 50 nm [21]. These aggregates are separated by macropores of the same size (or higher), and both, aggregates and pores, play the role of light scatters explaining the poor transparency of neutral 18% aerogel. The increase of TEOS concentration in the solution increases the numbers of clusters, and consequently their spatial extent is reduced. The cluster size decreases when the TEOS content increases because the growth of the clusters is limited by the neighboring ones. Therefore, for the highest concentration (46%) the size of the aggregates is lower than 10 nm [21, 23], favoring the aerogel transparency. In case of base catalysis, the range of aggregates size is lower than 10 nm [21, 22] which explains the high transmission in the visible range.

Measurement of pore size distributions by thermoporometry and N$_2$ adsorption-desorption techniques shows that the chemical parameters have an effect on the pore features. Pore size distribution is shifted towards the large pore size [16] when the organosiloxane concentration decreases in the gelling solution. When the base catalysis is used the mesopore size distribution is narrower [17]. So, base catalysis decreases the number of light scatters ( large clusters and large mesopores) leading to a better transparency.

Thanks to these results silica aerogels panel $30 \times 25 \times 45$ mm$^3$ (Figure 6) have been synthesized with TEOS concentration 18% and with ammonia aqueous solution in basic conditions (pH 8.5), the bulk density is $0.087 \pm 0.004$ g·cm$^{-3}$. The panel of two aerogels has been installed on the International Space Station (Figure 7).

After 18 months of revolution and return to the earth, the aerogels have collected several debris having different impact signatures and morphologies (Figures 8 and 9). After being retrieved from the space station, a wide range of impacts (0.1 mm) were observed in the aerogel from hard particles (metals) (Figure 8) or paint flakes (Figure 9). First results obtained on a small part of the aerogels indicate a large number of particles collected in the aerogels samples. We have found different materials: stainless steel, glass, mixed oxides…. This detector is able to collect very small particles from one to several μm. The complete study in terms of population, nature, and size of the debris is under progress.
5. Conclusion

Aerogel is an excellent hypervelocity space debris capture medium due to the fact that it is a highly porous material with a tortuous microstructure made up of nanoscale particles forming aggregates. When a debris or a particle impacts the aerogel as the aggregates are crushed, the debris is slowed and finally stopped (the tortuous structure of the aerogel dissipates its kinetic energy). Because of their peculiar physical properties: high porosity, transparency and tailored mechanical properties, base-catalyzed aerogels have been chosen to collect debris and microparticles in such a way to characterize the local environment of the International Space Station (ISS).

Aerogels, despite their relatively fragile nature, have survived to highly stressful conditions, and some debris have been captured intact and localized in the transparent aerogels. The following step will be to quantify and qualify the captured debris. The trace issued from the penetration in the aerogels will give information on their velocity and the associated shock. Taking into account the small penetration depth (lower than 10 mm) on Figures 8 and 9 and the density of the aerogels sensors, we can deduce that the velocity of the captured objects is lower than $5 \text{ km/s}^{-1}$ [4]. The complete analysis of the debris will allow an improvement of the models describing the space environment on low orbit and the associated damage risk.

References


