



Foaming unique composite cellular structures under microgravity by suborbital flight

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Abstract

The TESIS (TESTing In Space) experiment is a material science experiment carried out under microgravity during the Galactic 01 mission of the SpaceShipTwo Unity of Virgin Galactic on 29 June 2023. TESIS aimed to open suborbital flights to the approach of incremental research for the development of a new technology for medical applications. It consisted of 4 syringes, stored in the pockets of the astronauts and activated during the microgravity stage. The experiment was fully passive apart from the reaction of a small amount of liquid, separated into part A and part B of a polyurethane resin. Each syringe contained a foaming chamber and was confined into a vacuum-packed bag, tethered to the spacesuit of the astronauts. By pressing the syringe plunger, the resin parts entered in contact and started to foam. Fillers with different density (boron nitride and samarium cobalt) were added to the resin parts to produce composite foams during reaction. After sample recovery, the foaming chambers were extracted for non-destructive evaluation with radiological techniques. A comparison was made with on-ground foamed sample. Results show that the absence of gravity allows good mixing of the different fillers during the polyurethane foaming process. For the first time, composite foams have been produced in space using suborbital flight, and final achievements are very promising in the optic of in-space manufacturing of complex mixtures.

1 Introduction

This study combines the current trends in suborbital flights with the needs of technological research in microgravity and space environment. Experiment design and execution depend on the available opportunity to fly, from parabolic flights to long-term orbital missions. In 2014, the great potential of commercial suborbital space flights was already discussed [1]. It was noticed that these flights were expected to reach altitudes above 100 km, with 3–5 min of weightlessness. In fact, suborbital flights have characteristics in the middle between parabolic and orbital flights. The former are easy to implement, low-cost with very low and intermitted microgravity time. The latter are very expensive and difficult to implement, they require a lot of time for the experiment set-up and the access to a mission, but they provide very high microgravity times. The combination between the high frequency of suborbital flights and the passenger population was seen as a positive factor to open new scientific routes but only life-science research was mentioned, from passenger health and safety to basic studies of physiological mechanisms. The recent evolution of commercial suborbital flights has been driven by the increasing number of

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possible spaceflight participants [2]. At present, they have 2 dominant expectations: a view of the Earth from space and a weightlessness experience. According to the need of supporting space tourism, vehicles have been designed and optimized properly, as well as their trajectory [3]. This evolution of the future market for space tourists can become a fundamental opportunity for experimental research in microgravity conditions.

In cell biology, the effect of microgravity on cancer cells has been studied thanks to a parabolic rocket flight [4]. Some effects are discussed even if the minimum gravity time was just about 3 min. Because of this limitation, it is difficult to separate the effect of microgravity from all the others which could have influenced the cells, such as hypergravity, exposure to increased ionizing radiation, and extra stress during transport. Biological studies need a long time in microgravity conditions to be significant, because the cells, as well as any other living being, put time to adapt themselves to the new environment. Very recently, results on testing neuronal cells in microgravity by suborbital space flight have been discussed, thanks to Blue Origin's New Shepard NS-24 mission (on Dec 19th, 2023) [5]. Some differences have been found in comparison to control samples, left on Earth, and can be used to analyse possible neurological disorders and cognitive impacts of space travel.

Microgravity is not the only aspect of technical interests for suborbital flights, as they can be used also to measure the temperature profile of the atmosphere or to test shields [6]. For shields, a possible solution would be to include the payload in the interstage adapter of a rocket, and ejecting it during the ascent phase, thus performing up to 15 min of ballistic flight. This solution seems to be technically complex and expensive, and not applicable to payloads to retrieve.

Radiation is another aspect of interest for experiments in suborbital flights. However, in analogy with biological studies, the time of radiation exposure of a suborbital flight seems to be too low to investigate deeply the effect on biological structures. Nevertheless, some interesting data can be extracted also in this case. On 29 June 2023, the first-ever commercial suborbital flight of the Virgin Galactic (VG) SpaceShipTwo Unity was conducted and many research experiments were carried out [7]. Among them, a portable dosimeter-spectrometer, initially designed and manufactured for balloon flights, was used. The measured dose rates were in good agreement with previous data, from balloon flights in 2005 and civil aviation flights in 2015. It was concluded that there was a very small radiation risk for the pilots and astronauts flying in the SpaceShipTwo mission up to 85.11 km altitude. In a similar mission on June 8, 2024, on the same SpaceShipTwo of VG, an active personal wearable radiation dosimeter, namely IvmeRad-S was carried by a Turkish astronaut on the suborbital space flight up

to an altitude of 87,548 km [8]. For these cases, the use of a suborbital flight is an advantage in terms of fast checking of the instrument performance whereas mapping the local radiation dose rate seems to be less impacting. Besides, the environmental impact of suborbital flights is very high, and it should be avoided in the case of marginal research. It has been estimated that suborbital tourism releases 400–1,000 times more CO₂ per passenger per hour compared with commercial aviation flights [9]. However, the point of view should be the opposite. If space tourism cannot be interrupted, at least the maximum impact must be originated by associating experimental research.

Into this optic of the highest impact of space tourism on society, research of such physical mechanisms should be preferred as well as on innovative prototypes or manufacturing techniques. In the past, suborbital flights were already used for experiments on physical mechanisms under microgravity or minimum gravity conditions. In 2013, by the suborbital rocket flight REXUS 12, the collision properties of submillimeter-sized, highly porous dust aggregates were studied during a microgravity time of 3.5 min [10]. Results were affected by the sticking of the powders on the wall of the particle containers, and the accumulation of particles in a specific corner of the same containers. Nevertheless, researchers gained many useful indications to improve this kind of experimentation. This kind of study could benefit today from the increased number of available suborbital flights. They can become the optimal tool for incremental research in space environment. Incremental research involves step-by-step improvements, with small achievements on many consecutive experiments and continuous refinements. It is characterized by simple experimental procedures and fast testing, as the feedback from one experiment is fundamental to design the further. The value of the incremental research in aviation has been already discussed. Incremental steps have collectively transformed aviation research, such as the evolution of jet engines, winglet technology, and composite materials [11]. Incremental research has been never applied to traditional space experiments as they are typically very complex, time consuming and far one from the successive, when possible. Research on suborbital flights is at the conjunction between aviation and space; therefore, some revolutionary approaches can be originated by mixing these fields.

The rationale of the research implementation on suborbital flights is summarized in Fig. 1, in comparison with the other infrastructures for microgravity. Apart from drop towers, all the other infrastructures allow manned experimentation. Drop towers are not very expensive but they provide about 10 s of microgravity for a single drop, in the best cases. This time is doubled in parabolic flights, for a single drop. Physical mechanisms can be investigated into these

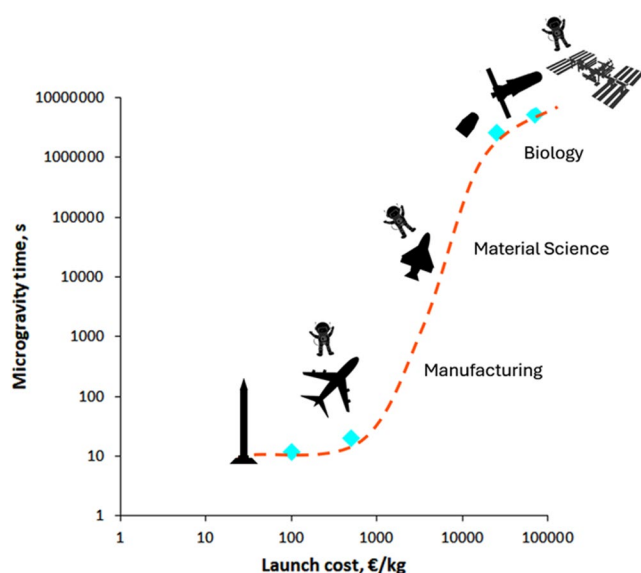


Fig. 1 Rationale of the use of parabolic flight in research

facilities which are used for initial studies before planning a long-term mission. In space, very long microgravity times are available at very high costs and with very low flight opportunities. Apart from biological studies, microgravity times are also too long for the needs of such experimentation and are optimized by repeating tests or testing different configurations. In the middle, suborbital flights can be used for better understanding the mechanisms or the systems to test in long-term missions, or to make long experimentation with the concept of the incremental research. This approach would open suborbital flights to the experimentation on material science and manufacturing. The current study is the example of the application of this approach to liquid foaming, which is at the conjunction between material science and in-space manufacturing. The potential of microgravity on liquid foaming has been already shown in the past, but related studies are fragmented because of the difficulty to make tests in space. Probably, less than 10 foaming experiments have been performed up to now, by considering very different cases, from polyurethane resins to aqueous solutions and metal foams [12–18].

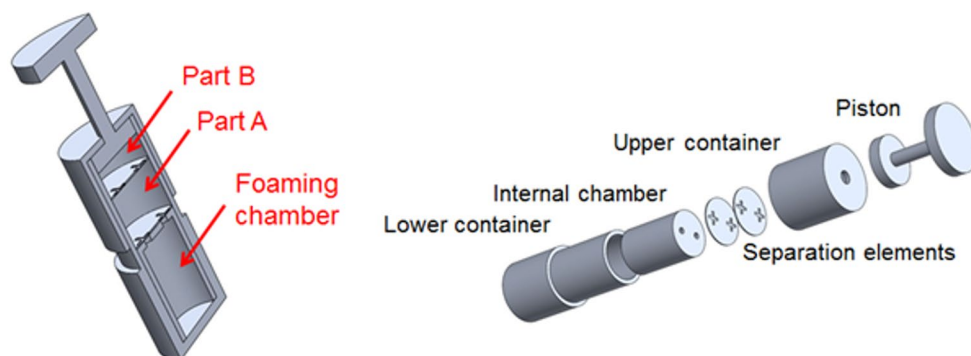
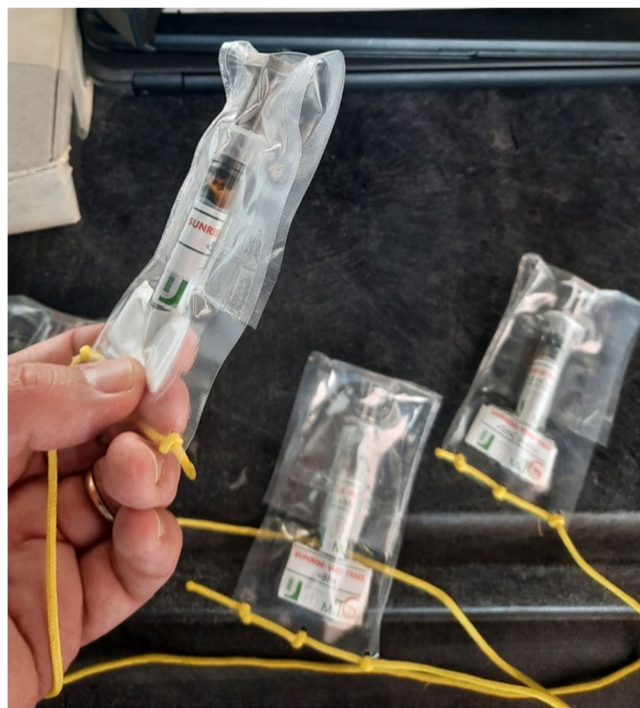
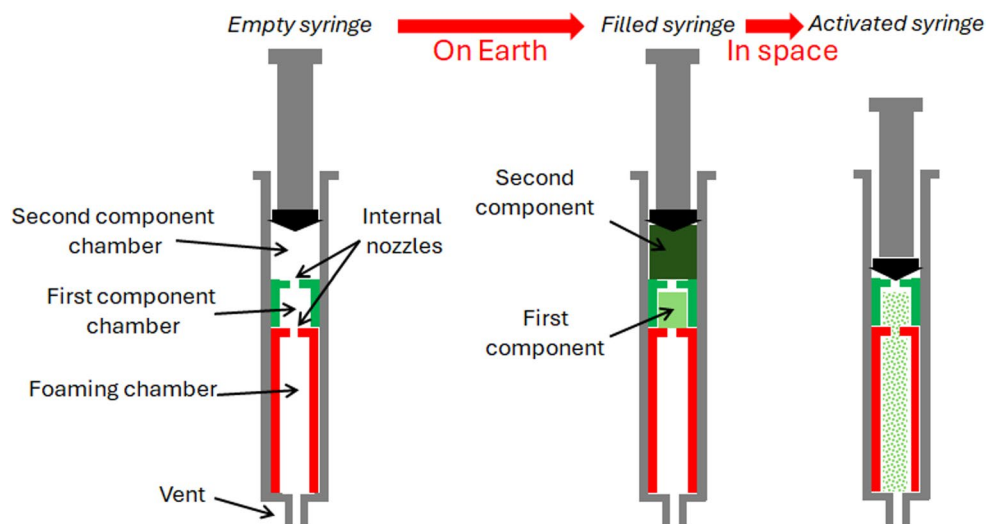
Thermosetting polyurethane foaming was investigated under microgravity, but available studies are few and old [12–13]. The reason is that those studies focused on the evaluation of the effect of gravity on the basic mechanisms of the foaming process, rather than on the way to use this unique environment to overcome technological limitations of current manufacturing technologies. In fact, foams were all pure. The best contribution has been provided in 2000, as it was found that PU foams formed in microgravity have near perfect structures with smaller cells than those of under gravity [12]. Experiments were made by using Consort series research rockets [13]. By considering also other foam

typologies, in 2011, thanks to the ESA FOAM project, it was observed that foams containing large amounts of liquids are stable in microgravity for longer time than on Earth [14]. However, it was discussed that foam generation can still be limited [15]. In 2011, results of the FOCUS experiment were also analysed [16]. Foams containing particles were fabricated in microgravity by using a special foam generator with an aqueous suspension of silica nanoparticles. Experiments in microgravity can be extended far beyond the same made on Earth [17]. Liquid metal foams have been studied as well through X-ray radioscopy during parabolic flights [18].

According to the possibility of flying in a suborbital flight, a simple experiment architecture was developed. Combining material science with manufacturing innovation, this study discusses the results of 4 different foaming experiments performed by the Italian astronauts on the first-ever commercial suborbital flight of the Virgin Galactic (VG) SpaceShipTwo Unity (on 29 June 2023). The experiment was named SUNRISE-VG02-TESIS by merging the acronym SUNRISE (Second University of Rome In Space Environment), with VG (Virgin Galactic) and TESIS (TESTing In Space) whereas the number 02 referred to the fact that a second experiment was also performed in the same mission (named SHARCS, SHape Recovery of Composite Structures). For TESIS, the astronauts activated fully passive payloads to manufacture, in space, composite foams which are difficult to produce on Earth. Radiological tests have shown the unique properties of these composite foams in terms of homogeneity and cell structure.

2 Materials and methods

A simple experimental apparatus has been developed to allow polyurethane foaming in microgravity according to the scheme of Fig. 2. The system consists of a piston that can slide at the top of a cylindrical chamber up to touch the upper surface of a bottom chamber. At the top, there are 2 volumes, separated by a diaphragm, in which the 2 parts (A and B) of a liquid polyurethane resin can be inserted without contact. A diaphragm separates also the volume of part B with the bottom chamber, which is initial empty, called foaming chamber. By pressing the plug, the upper diaphragm breaks and the two resin parts go in contact, starting reaction. Contemporarily, their mixture flows into the bottom chamber by breaking also the second diaphragm. This configuration has been designed to allow maximum mixing in a short time, in absence of gravity. In fact, mixing is achieved by the shear and extensional flow of the resin mixture through the holes of the indented diaphragms. It is not necessary to reach the full polymerization during the

Fig. 2 Conceptual design of the TESIS payload**Fig. 3** Engineered TESIS payload**Fig. 4** TESIS payload before flight

microgravity stage, just the gelation to avoid that the following accelerations, from the spacecraft vehicle, would modify the foam structure. It is, instead, fundamental that the plug is actioned under microgravity.

2.1 Payload engineering and manufacturing

Due to limitations from the available time for experimentation and the safety of the operations in presence of astronauts, the TESIS payload was designed as a wearable apparatus and divided in 4 parallel and independent tests. Therefore, the conceptual design of Fig. 2 was engineered in the apparatus of Fig. 3, where a common syringe for medical uses has been adapted to contain the resin and foaming chambers. The hollow tip of the syringe was used as vent, whereas the foaming chamber and the Part A chamber were 3d printed in the shape of hollow cylinders, with a restriction at their top. The part B volume, instead, was directly confined by the syringe internal wall, thus allowing the plunger sliding. Thin thermoplastic sheets were placed at the top of the 3d printed cylinders to work as diaphragms.

In Fig. 4, the final syringe is shown in its envelope, sealed under vacuum. An adsorber was also added at the tip of the syringe to collect possible excess of resin during foaming.

Table 1 Composition of the syringes of the TESIS payload

ID	Matrix, %wt	Filler 1	Resin part	Filler 2	Resin part
1	100	No		No	
2	97	BN,	A	No	
3	88	Sm ₂ Co ₇	A	No	
4	85	BN	A	Sm ₂ Co ₇	B

A tethering rope (in yellow) was added to guarantee safe constraint of the payload to the astronauts' suite. The initial syringe had a 2.5 ml capacity, and 4 syringes were prepared with different compositions (each one with a final weight about 6 g). The composition was changed by adding fillers in the part A and part B of the resin. The resin was a 2-component polyurethane, RP491A ESPAK 90 by Prochim srl. The ratio between polyols (part A) and isocyanates (part B) of the polyurethane mixture was 1:1 vol, and the full amount of resin for each payload was 0.15 ml. The composition of the 4 syringes for the on-orbit experiment is shown in Table 1. The first syringe was filled with part A and part B of the neat polyurethane. In the second and third syringe, boron nitride powder (BN, by Sigma-Aldrich) and samarium cobalt powder (Sm₂Co₇, by Sigma-Aldrich) were added as fillers in the part A. In forth syringe, both powders were added, one in part A and the other in part B of the resin. These fillers have been selected for their very large difference in density and their potential use for radiation shielding [19, 20]. In fact, samarium cobalt has a density of about 8.3 g/cm³ and tends to sink in the liquid resin under gravity, without any possibility to disperse into the cellular structure during foaming. Boron nitride is 2.1 g/cm³, much less than Sm₂Co₇ and closer to the liquid resin but its fine powder is hard to disperse as well. Their combination into the same liquid resin would request complex mixing on Earth, also in absence of a foaming step. In the experiment design, it was planned to reach the nominal composition of 3 wt% for BN and 12 wt% for Sm₂Co₇ in the final foam, with a ratio 1 to 4, the same ratio of the filler densities. The single filler foams had the same weight of BN or Sm₂Co₇ into to the 0.15 ml of resin, therefore the filling content increased from 85% of the combined foam to 88% of the Sm₂Co₇ filled foam, and 97% of the BN filled.

The TESIS payload was designed to be fully passive, to minimize its impact on the mission and to simplify their use by the astronauts. Being wearable, they were put into the spacesuits, in the pocket on the arm. Being three astronauts involved in the experimentation but having 4 syringes, one of them had to activate 2 syringes.

2.2 TESIS experiment

On 29 June 2023, the TESIS experiment was carried out at the start of the microgravity period, to maximize the

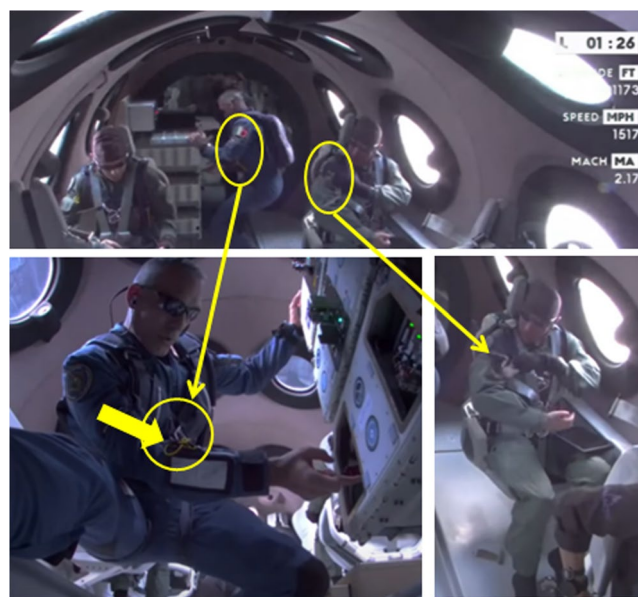


Fig. 5 The TESIS experiment during the microgravity stage of SpaceShipTwo Unity (courtesy of Virgin Galactic)

time for the resin gelification before the vehicle re-entering (about 3 min). In Fig. 5, an image is shown during the SpaceShipTwo Unity flight in the microgravity stage. An astronaut is visible who is placing back the syringe into the spacesuit pocket, whereas the yellow tethering rope of the payload is visible on the arm of another astronaut who is working on other experiments of the mission. The TESIS experiment was executed fast and safe, as expected, with no impact on the other experiments of the mission. In the end of the mission, the payloads were immediately available for testing. For comparison, other 4 syringes were manufactured and activated on ground to take under consideration the effect of the gravity.

2.3 Testing

After recovery, the syringes of the TESIS payload were open to extract the foaming chamber, together with the syringes activated on ground. All the syringes were correctly activated even if some samples were better foamed than others. First tests were carried out without affecting the sample integrity. For this aim, a radiological evaluation was carried out to study the distribution of the particles contained in the foams. The plastic foaming chambers were placed it on a nearly radiolucent foam support vertically. Radiological evaluation was performed using a 128-slice CT scanner (GE Medical System, Revolution CT). The CT parameters were defined considering the small size of the particles: 80 KV voltage, 100 mA current, X-ray tube rotation of 1.00s, layer thickness of 0.6 mm and a gap of 0.531:1. After acquisition, colorimetric maps were obtained

Fig. 6 Vertical section of the foaming chambers of the TESIS payload and the on-ground foamed samples by the radiological analysis

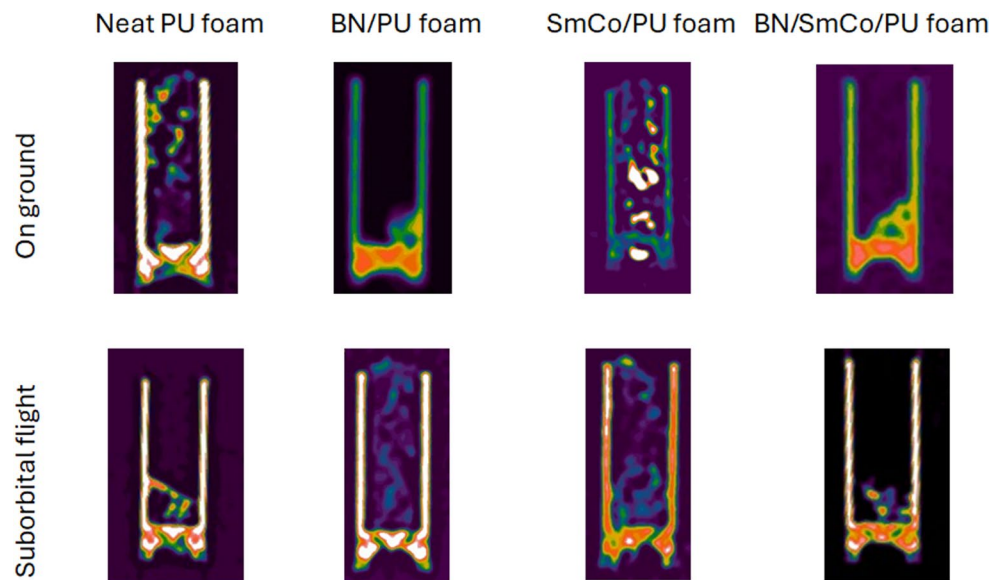
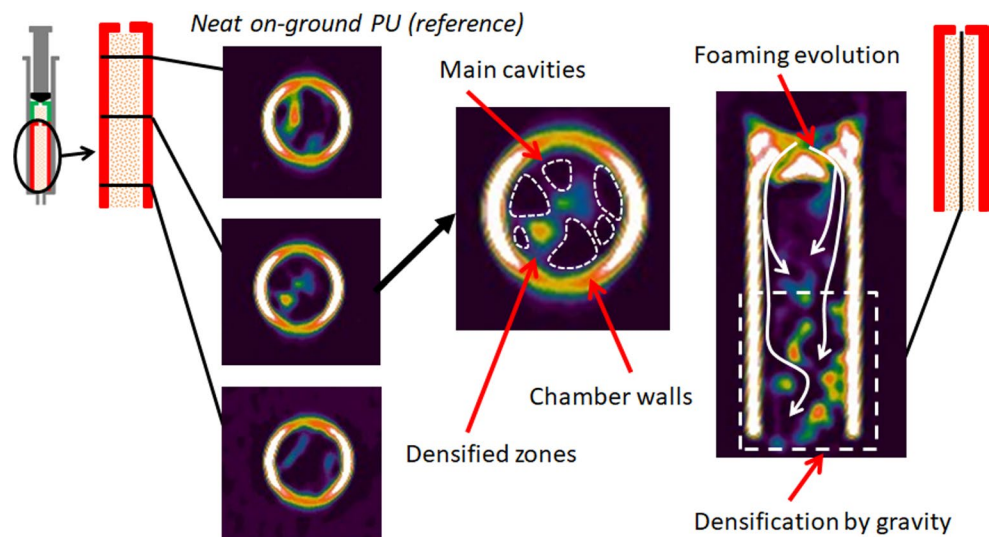


Fig. 7 Axial and transverse sections of the neat polyurethane sample foamed on-ground



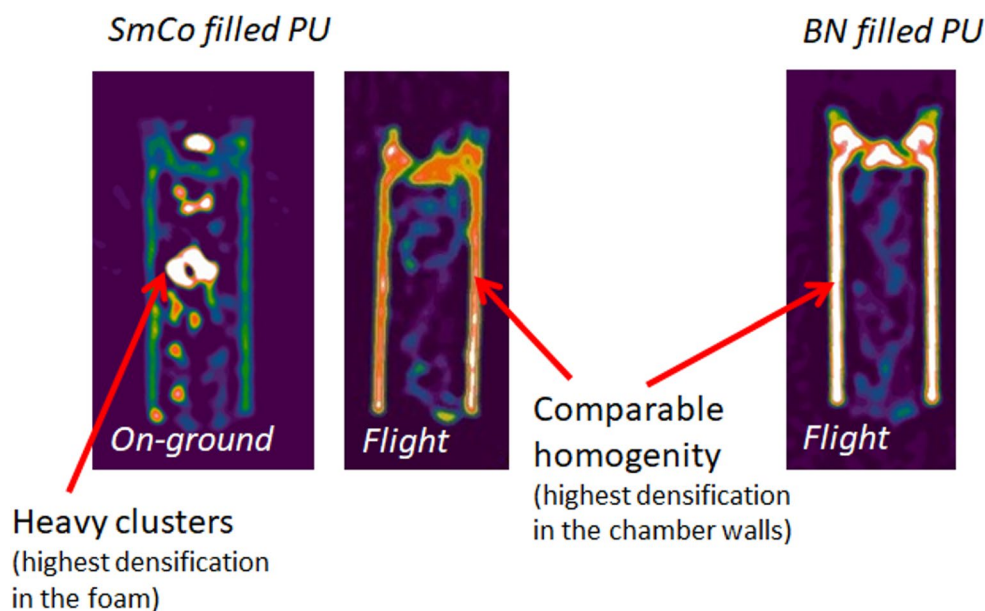
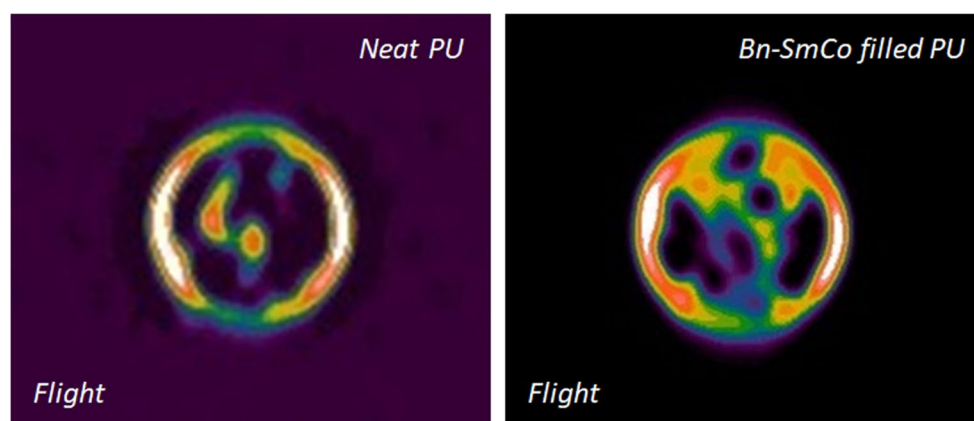
and a virtual reconstruction was generated to navigate the samples entirely and to study the aerial caveolae.

3 Results and discussion

A comparison between the samples foamed under microgravity and on-ground is shown in Fig. 6 where the colorimetric map of the foaming chamber is shown at its maximum section. It is visible that syringes with single filler foamed very well in microgravity whereas the other 2 syringes foamed partially, comprising the neat polyurethane. This result could depend on the different sensibility of the astronauts who activated the syringes or on possible malfunction. It is a common risk of incremental research when the testing apparatus must be very light and simple. In fact, this risk was managed by incrementing the number of

syringes up to 4 and involving different astronauts. Thanks to this approach, very important results have been obtained and are discussed, for the first time, into this study.

The results of the on-ground foaming experiments were expected. The neat polyurethane resin foamed correctly and fully filled the chamber. The final structure shows the typical morphology of the foam with a distribution of cell size. Figure 7 shows some axial sections of the neat sample foamed on-ground. It is visible that some densified zones are present, adjacent to large channels, which seem to correspond to the foaming evolution. Large cavities are present, in contact with the chamber wall. Densification and cavity growth may depend on the effect of gravity, at the end of the resin flow. In fact, during foaming, the gas produced by the reaction, for the bubble formation, provides structural support to the cells. When the gas pressure is released, the bubbles can collapse into larger cells and densified regions.

Fig. 8 Comparison between flight and on-ground samples**Fig. 9** Similarity of the filled sample foamed under gravity and the neat polyurethane foamed on-ground

Unfortunately, a full comparison with neat polyurethane in microgravity is not possible because of the small extent of the foaming step for the flight sample. The same problem has been observed for the syringe with both fillers. Into the current experimentation, as the foaming chambers have not been opened and only non-destructive evaluations have been performed, a comparison with on-ground foamed samples is only partially possible. In the future, the foaming chambers will be cut, and microscopic analyses will be carried out also in the component chamber. In that occasion, new findings could be discussed.

Differently from flight samples, bad foaming of on-ground samples with BN has been observed on several tests. In all these cases, the fine BN particles remained separated by gravity from the liquid resin, making difficult the foaming process. A similar effect has been observed also in presence of samarium cobalt, but in few cases, as that of Fig. 6, foaming occurred by isolating large clusters of particles. For this reason, the best comparison between flight and

on-ground samples is provided in Fig. 8 where the samples with single fillers, foamed in microgravity, are compared with the case of the on-ground foam with samarium cobalt. It is evident that clusters are absent in the flight sample with Sm_2Co_7 . Density distribution is very homogeneous, better also than the neat polyurethane foamed in presence of gravity. The homogeneity is also comparable with the other sample, filled with BN and foamed under microgravity. The similarity between the cell morphologies of the flight and on-ground samples is also shown in Fig. 9 in the case of the foam with both fillers. Despite of the small foaming extent, this analysis suggests that microgravity can partially nullify the effect of the powders on foaming, by reducing clustering effects. However, this consideration must be validated by further studies when the foaming chambers will be cut to perform microscopical and spectrum analysis. At this stage, the integrity of the samples has been preserved to deepen the analysis on the foam morphology. In fact, there is the risk, partially confirmed in the laboratory practice, that the

foams have stuck on the chamber wall, and could be irreparably broken during extraction.

4 Conclusion

In the TESIS experiment, for the first time, a foaming process was carried out in the microgravity phase of a suborbital flight, thanks to the adoption of a unique wearable device. Moreover, for the first time a composite foam was successfully foamed in absence of gravity. The main goal of the TESIS experiment was studying the effect of microgravity on mixing, in the optic of future in-Space manufacturing for technical and biomedical purposes. The focus is developing new manufacturing routes for complex foams. Blending a mixture of powders into a homogeneous system is a crucial step not only in medicine and pharmacology but in many manufacturing processes [21]. Production of foams with materials with very different density is a challenge, as these foams are very difficult to produce on the Earth. Secondary goals of the experiment were the evaluation of the behavior of binary systems in microgravity, the effect of microgravity on liquid-solid mixing, the set-up of quick tests for future studies in suborbital flights, the evaluation of the impact of experiments in suborbital flights for the research into this field.

Composite foams with polyurethane base have been manufactured under microgravity, according to the logic of incremental forming. Very simple experiments have been planned and carried out with the risk of partial success of such operations. Nevertheless, by increasing the number of experiments, with a robust conceptual approach, it is possible to achieve unique results. In the current experimentation, 2 syringes on 4 had optimal foaming, for the other 2, additional testing is necessary with sample destruction to deeply discuss possible new achievements. However, by using non-destructive evaluation techniques on the best foamed samples, with single filler, the effect of microgravity is evident. Filled foams are homogeneous in density despite the different fillers (BN which is light and Sm_2Co_7 which is heavy). Clusters are also absent in foams with high density fillers. By comparing with samples foamed on-ground, it is possible that such gravity effects on densification have disappeared. Moreover, in all the flight samples the highest densification is found in the chamber walls.

In the case of the complex BN/ Sm_2Co_7 mixture, similar morphology and densification of the on-ground foamed neat PU is found. This is a very interesting achievement, which strengthens the interpretation about the similarity in the morphology of single filler flight samples. The absence of gravity is able to produce very fast and very effective mixing of liquids also in the case of strong differences in the

substances to mix, in terms of density, size distribution of the particles and viscosity of the matrix.

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Author's contribution All authors contributed to the study conception and design, material preparation, data collection and analysis. The first draft of the manuscript was written by Fabrizio Quadrini and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Data availability The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Competing interests Authors declare they have no financial and competing interests.

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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