Synthesis and Characterization of Silicon-Based Thermal Protective Syntactic Foams

Clara-Ann Cheng Ling[1], Kelly Siah Xinjie[1]
Yap Jing Han[2] (yjinghan@dso.org.sg), Dai Jie[2] (djie@dso.org.sg)
[1]Raffles Girls' School (Secondary), 20, Anderson Road, Singapore 259978
[2]DSO National Laboratories, 20 Science Park Dr, Singapore 118230

Abstract— This project explores silicon-based thermal protective materials. In this project, potential silicone resins and colloidal silica will be screened, with suitable ones identified as the matrix. To impart thermal resistance and thermal insulating functions, various inorganic fillers will be studied and synthesised with the silicon-based matrix to formulate such thermal protective materials. Various glass bubbles of the 3M S and K series will be incorporated into the matrix resin, along with different filler to resin to glass bubbles ratios in order to obtain an optimal thermal protective material that will function as a thermally stable and insulating layer to protect inner substrates to withstand thermal exposure. Syntactic foam synthesis as well as curing behaviour will be monitored to determine the best composition.

1 Introduction

This project aims to develop a syntactic foam that is able to act as a thermal protective material with low density, low mass loss under high temperatures, and high compressive strength. 2 types of silicon-based syntactic foams were synthesised and tested: silicone-based syntactic foams and silica-based_syntactic foams. To impart thermal insulative properties to these foams, hollow glass bubbles were dispersed through the matrix during synthesis, thereby creating small air pockets within the matrix of the syntactic foam. These air spaces reduce the rate of heat transfer through the syntactic foam. Syntactic foam synthesis as well as curing behaviour will be monitored to determine the best composition.

2 Materials and Methods

The type of microsphere, microsphere concentration, and resin are found to affect mechanical properties of syntactic foams.

Firstly, resin selection takes place, from a list including HTSB polymers and commercially available colloidal silica Ludox HS40. After curing at room temperature and at 250°C, each syntactic foam will be burned at 600°C and 800°C. Little shrinkage and no cracks when curing or burning will indicate a plausible potential resin. After eliminating resins that have warped, shrank, or cracked, the percentage mass retention of each syntactic foam will be analysed to choose a resin that retains most of its mass at high temperatures. After which, a filler will be incorporated into the silicon-based matrix. Four different fillers are tested, namely mica, kaolinite, titanium dioxide and metakaolin. Similarly, through the same criterion, a suitable filler will be selected. This is repeated varying the S and K series of HGB. Lastly, percentage composition of the silicon-based resin will be varied.

Percentage mass retention as well as stress at upper yield of the room temperature sample under compression will serve as criteria to derive a good formula composition that yields a viable silicon-based material that is able to withstand high temperatures with little to minimal warpage and shrinkage.

3 Results – Thermal Conductivity

In syntactic foams, a large factor affecting thermal conductivity is density. As the conductivity of the silicon-based matrix is specific to the type of resin or ceramic formed, the density of the foam was controlled by changing the proportions of S38 to K1 HGB. S38 HGB have a density of 0.38g/cm^3, while K1 HGB have a much lower density of 0.125g/cm^3. This is due to the structural differences between both types of HGB. K1 HGB have a larger particle size as well as a thinner glass wall, leading to a much larger air space within the sphere, thereby decreasing the overall density of the foam when the ratio of K1 to S38 HGB is increased.

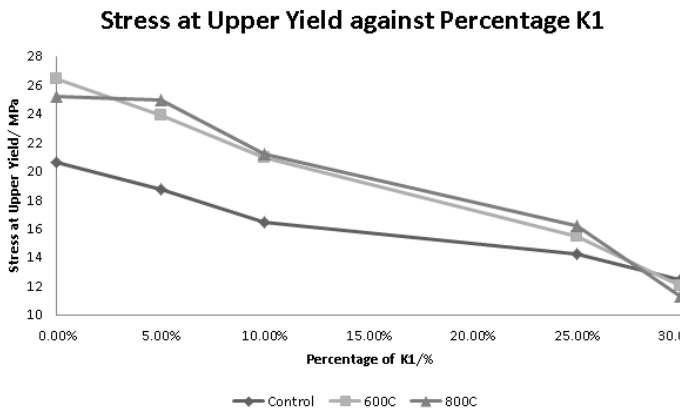

Fig. 1: Stress at upper yield against percentage K1 graph

While as the percentage of HGB in the syntactic foam that are of the K1 variety increases, the density decreases, and may therefore create a syntactic foam that has a lower thermal conductivity, the resulting syntactic foam is found to be less able to withstand compressive strength. As shown in Figure 1, as percentage of K1 HGB increases, stress at upper yield decreases as well for the silica based resin. This is because K1 HGB are less able to withstand compressive strength than S38 bubbles.

At low percentages of K1, the compressive strength of samples which have been heated at 600°C and 800°C are higher than the control group, as shown in Fig 1. This is likely due to sintering occuring at those temperatures, albeit to a small extent. This would give the entire structure ability to withstand higher compressive load. However, as the percentage of K1 HGB increases, the compressive strength of the samples which have been heated decreases in relation to the control group. At 30% K1 HGB, the compressive strength of those which have been heated is lower than that of those which have not been heated.

This is likely due to the weaker mechanical properties of K1 HGB. K1 HGB, having thinner glass walls, warp and deform more easily at high temperatures of about 600°C. When put under compressive load, these HGB fracture and collapse more easily, thus rendering the entire syntactic foam less strong under compressive load. This is coupled with the fact that large K1 glass bubbles may hinder the sintering process, and as such the syntactic foam does not gain compressive strength due to sintering.

4 Results – Mechanical Testing

Results from the LR30X PLUS UTM machine are plotted in Figure 2. The compressive strength of all samples were plotted against the stress at upper yield values obtained from the stress strain curves.

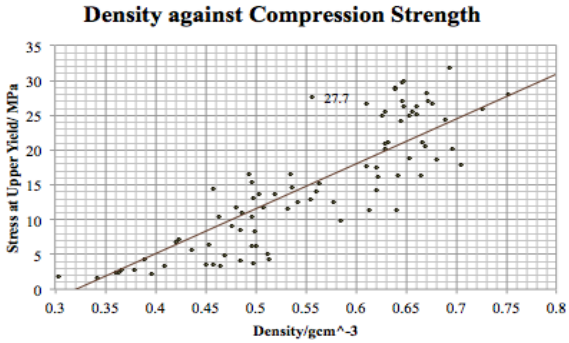

Fig.2: Density against compression strength graph

In Figure 3, a clear comparison is drawn between silicone-based and ceramic-based syntactic foams. Cells highlighted indicate properties desirable for the purposes of thermal protective materials.

Silicone-based syntactic foam	Ceramic-based syntactic foam
Lower density	Higher density
Lower compressive strength	Higher compressive strength
No/little warping/shrinking	Significant warping/shrinking
Higher mass loss of 4-5% at 600°C	Lower mass loss of 1-2% at 600°C
Turns into silica ash at higher temperatures (>1000°C)	Sinters at higher temperatures, increasing compressive strength further, but also increasing brittle nature

Fig. 3: Table showing comparisons between silicone-based and ceramic-based syntactic foams

Using ability to cure without shrinking or warping, mass loss at 600°C and 800°C, compressive strength and density as criteria, the most suitable silicone-based syntactic foam and ceramic syntactic foam are selected, and the optimal compositions are determined.

4 Conclusion

The results of experimentation serve to reveal the factors that are related to compressive strength of syntactic foams. As the density of the syntactic foams increases, the compressive strength of the syntactic foams increases. The type of HGB used in the syntactic foam also has a major impact on the compressive strength of the syntactic foam, with HGB which have thinner walls and larger particle size causing the syntactic foam formed to have a lower compressive strength.

The potential applications of such silicon-based syntactic foams are immense - from use as structural components in space travel vehicles to marine equipment in deep sea operations.