Technical Insight

Title: Temperature limits of functionalized surface coatings
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Background:

This technical insight is focused on our functionalized coatings of SilcoNert® 2000 and Dursan®. Each has a monolayer deposition that occurs after the coating is deposited to tailor the surface properties. This surface functionalization will degrade at high temperatures. To measure the temperature at which the surface functionalization is lost, we used a thermogravimetric analyzer combined with a mass spectrometer to identify any evolved gasses.

Goal:

To provide data to support the upper temperature limit of SilcoNert® 2000 and Dursan®. These temperature limits could change depending upon the chemical environment that the coating encounters, so both an inert atmosphere of helium gas as well as an oxidative environment of air were used for testing in this technical insight.

Discussion / Data / Links:

Experimental
To monitor the loss of the surface functionalization, a TGA Q50 from TA Instruments was used. The TGA is a very sensitive balance that has a resolution of 0.1 µg, and both helium and air are available for creating an inert and oxidative environment. It is attached to a Pfeiffer Vacuum mass spectrometer to monitor any gaseous species that is evolved from the coatings.

Typically, our coatings are applied to low surface area pieces such as tubing and flat surfaces. As sensitive as the instrument is, the small amount of surface area, and thus surface functionalization, is not an ideal situation for measuring evolved gasses. For this reason, coating a high surface area material such as a powder was necessary to create enough coating for the instrument to detect the small changes in mass as well as the gasses that are evolved. We used 5 µm silica particles as the substrate in this study.

Particles coated with SilcoNert 2000 and Dursan were heated to 900°C at a rate of 20°C/min. The mass spec was used in "bargraph" mode which surveys across numerous chemical species. In our experiments, mass to charge ratios (m/e) of 4 to 80 were monitored. Unfortunately, the mass spec used here is unable to monitor any m/e below 4, which would be useful in monitoring hydrogen evolution. Both SilcoNert 2000 and Dursan were run in a helium and air environment to demonstrate an inert and an oxidative environment.

Results and discussion

SilcoNert® 2000, which has a functionalization of to the surface of hydrogenated amorphous silicon showed that when the mass started to drop, there was a release of m/e = 25 through 30 which is representative of a combination of components of the functionalization. Figure 1 and Figure 2 are shown with an m/e of 30.

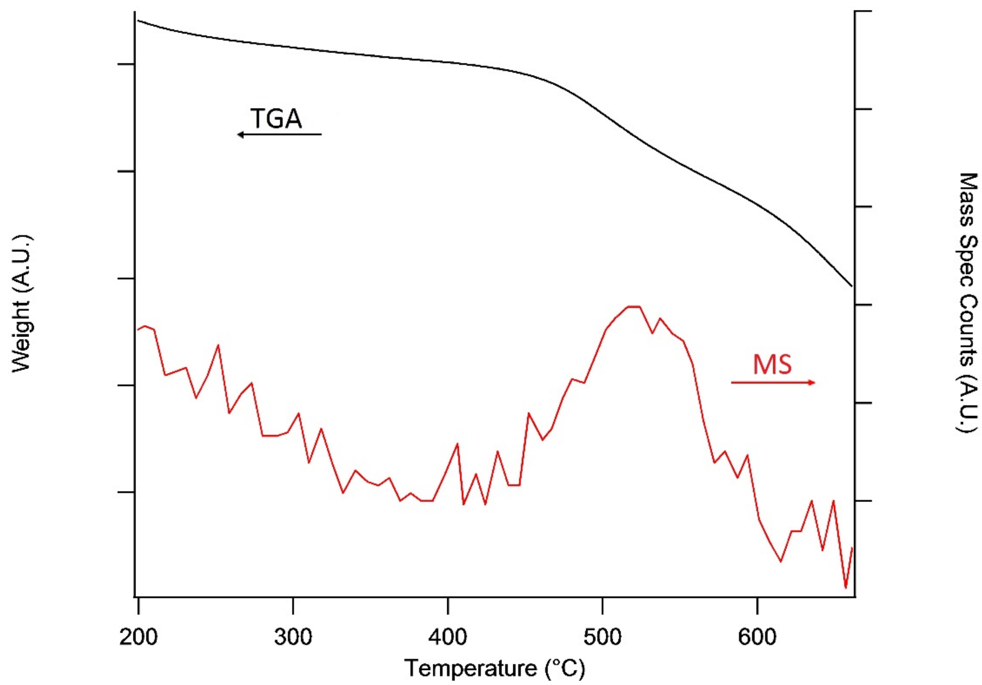

Figure 1: TGA-MS of SN2000 which was run in an inert atmosphere (Helium).

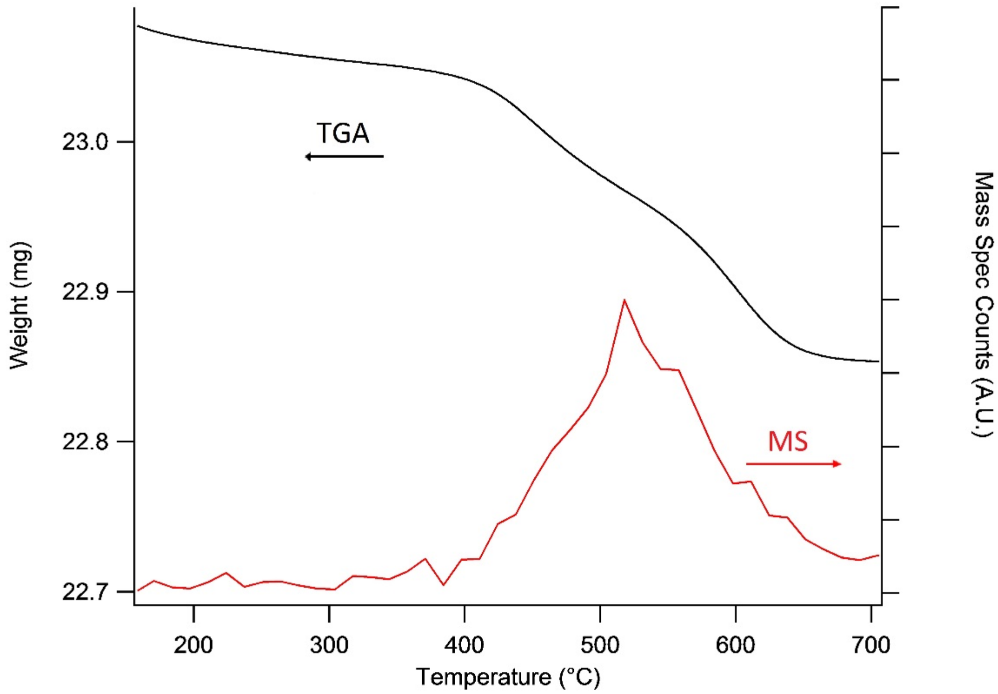

Figure 2: TGA-MS of SN2000 which was run in an oxidative atmosphere (air).

The surface functionalization loss starts at about 450°C for the inert atmosphere and 400°C for an oxidative atmosphere. The TGA data in both shows what appears to be two steps in weight loss. The first is associated with the functionalization leaving the surface. Since there was no detectable signal from the mass spec, the second weight loss can be assigned to the loss of hydrogen in the amorphous silicon base coating. These results closely match other reports of silicon surfaces that were functionalized via hydrosilylation.[1] Based on this, it is reasonable to assume that other silco-coatings such as SilcoKlean® also start to lose their surface functionalization around these temperatures as well.

The Dursan® surface is functionalized to enhance the performance (functionalized coating is called Dursox™). During the experiment a variety of gases are released as well as larger masses such as an m/e = 44 which can be assigned to an $SiCH_x$ species. These species start to evolve from the coating at the same temperature, and in Figure 3 and Figure 4, the m/e of 44 is shown. The onset of surface functionalization loss in an inert atmosphere is around 500°C and in an oxidative atmosphere it is 450°C. As was seen in the SilcoNert 2000 example, Dursan appears to also have a 50°C shift in the loss of surface functionalization when run in an inert environment rather than an oxidative environment. There is also a noticeable second peak in the mass spec data at a higher temperature. This is a result in additional gasses being lost from the bulk of the Dursan coating rather than just the loss of the surface functionalization. This will be investigated further in a future technical insight.

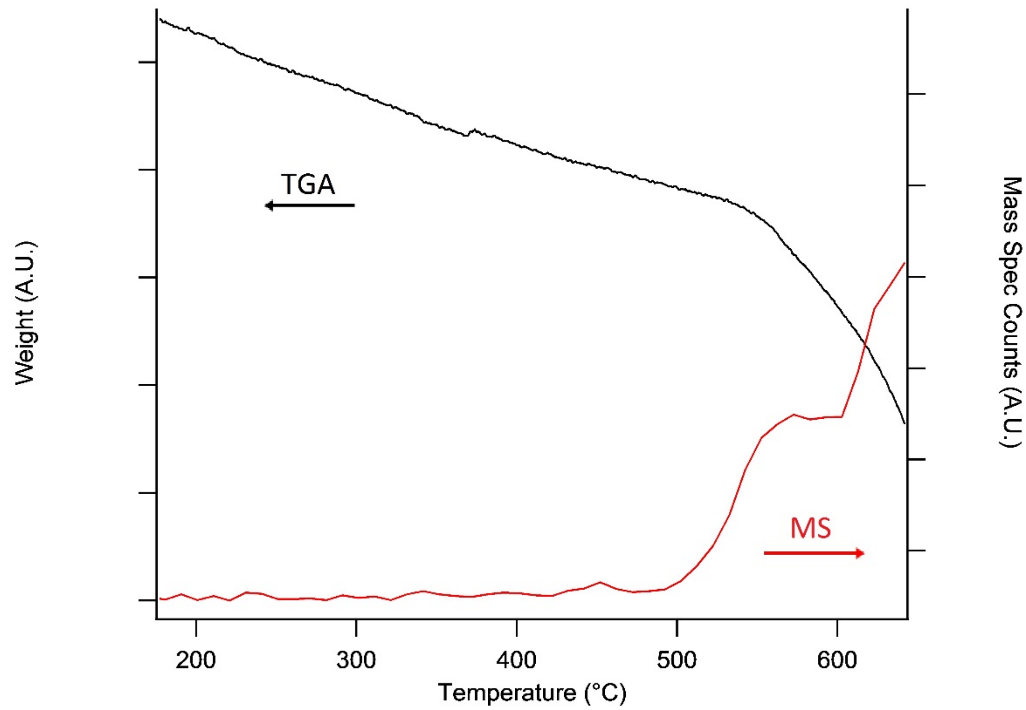

Figure 3: TGA-MS of Dursan which was run in an inert atmosphere (helium).

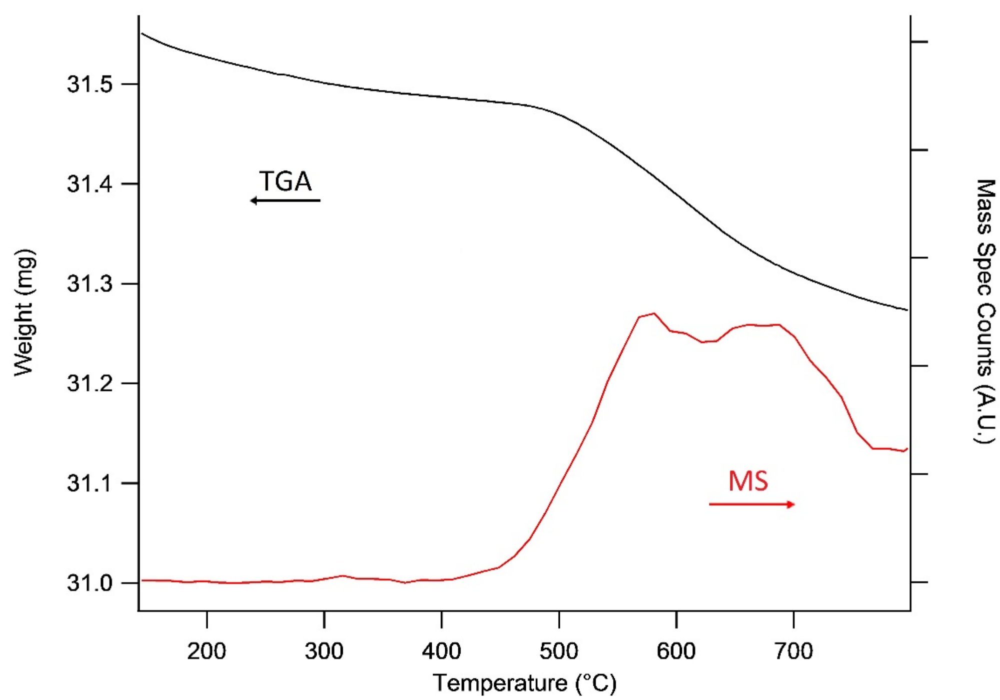

Figure 4: TGA-MS of Dursan which was run in an oxidative atmosphere (air).

Conclusions

The current coating specifications for SilcoNert® 2000 and Dursan® are 450°C. This study shows that the number is accurate for SilcoNert 2000 in an inert atmosphere, but it is closer to 400°C in an oxidative one. Dursan's surface functionalization can withstand a higher temperature of 500°C in an inert atmosphere and 450°C in an oxidative one.

Some customers do use these coatings in environments that exceed these temperatures. In these cases, it is important to understand their application and use of the coating. If they are using the coating for its inert properties, after the surface functionalization is gone, they will still have SilcoNert 1000 or Dursox remaining. These coatings are also inert, but not to the level of inertness that can be seen in the functionalized coatings, and they may see a degradation of inertness over time.

References:

1. Dahmen, C., *et al*. "*Surface functionalization of amorphous silicon and silicon suboxides for biological applications*". Thin Solid Films. **427**, (2003), 201-207.