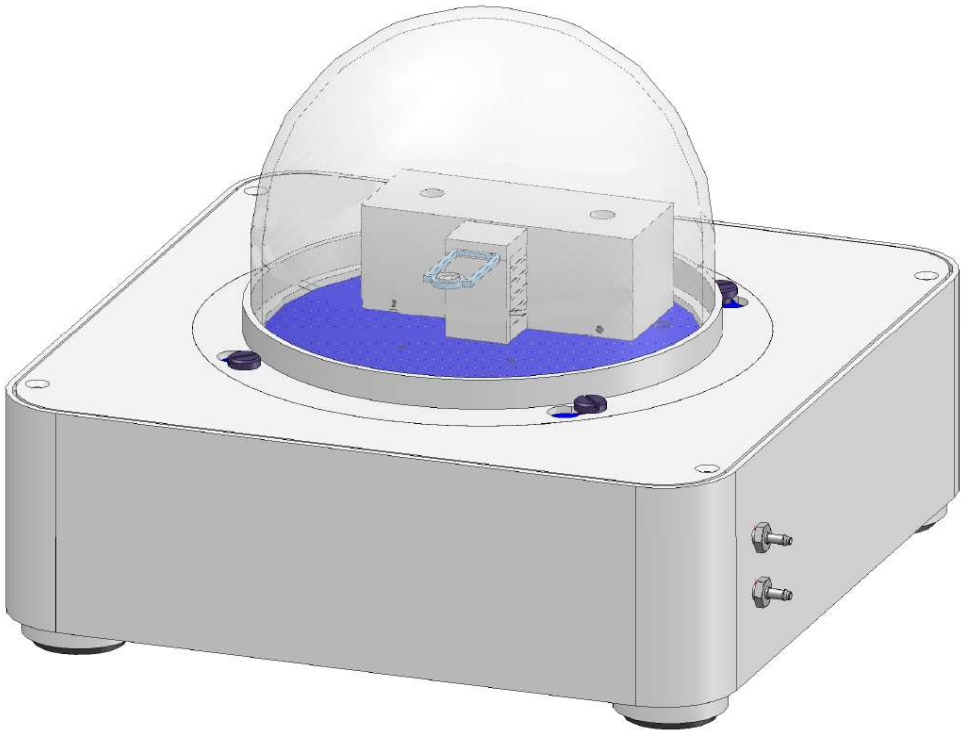
How To

Sample preparation

with Chip DSC



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Stand: 17.03.2020

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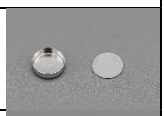
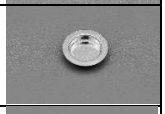
1. General information

This manual is a short description of how to handle and prepare samples for the Chip DSC. Generally better results are obtained when the sample is in intimate contact with the bottom of the sample crucible. Sample preparations that improve this condition will help transfer heat between the sample and crucible and give more heat signal to measure. This is particularly important for measuring phase transformations that have low endothermic / exothermic energy properties. For reversable process better result might be achieved during a second heating cycle as contact intimacy improves. Another area of importance is what type of crucible should be used for a specific application. This document also gives some guidance for crucible selection. Most DSC investigations will require some methods development for preparing the sample for optimium results. For more Information, read the other available instructions about software or specific manuals for the Chip DSC.

2. Choose the right crucible

To be sure you use the right crucible for your needs you should check what type of sample you want to measure. There are serval types of crucibles for Chip DSC

Name	Best.-Nr.	Description	Picture
Al Standard crucible 6x1,5mm 40µl	30293042	The most lightweight crucible and easy to use	
Al crimpable crucible 100µl	30293043	The biggest crucible for voluminous samples. Crimpable to measure with atmosphere	
Al crimpable crucible 40µl	30293045	The small crimpable crucible to measure with atmosphere	
Al lid without hole	30293044	Compatible with 30293043 and 30293045	
Al lid with hole	30293046	Compatible with 30293043 and 30293045	
Al crucible for foils with lid	30293050	Crimpable crucible to measure foils and powders. Special crimping tool is needed.	
Al crucible for small samples with lid	30293051 30293052	Crimpable crucible to measure small samples. Special crimping tool is needed	
Cu crucible	30293049	The copper crucible to measure Oxidation induction time and Oxidation onset temperature	
Other crucibles (Al_2O_3, Graphite, Platin, aso.) available on request			
Request the compatible crimp-tool for your crucible at your supplier or sale@linseis.com			

3. Crimp crucibles

3.1. Crimpable Crucibles and lid without hole

Depending on your sample you want to measure it could be necessary so measure under inert gas atmosphere. In this case you should use crimpable Al crucible with a lid. You can use them up to 0.1 MPa.

3.2. Crimpable Crucibles and lid with hole

If you want to avoid your samples to spread on your sensor and out of the crucible, you can use the lid with hole on our crimpable crucibles

3.3. Al Standard Crucibles 6x1.5 mm 40µl

To do fast measurements the easiest way is to use our 6x1.5 mm standard crucible. It is very lightweight and flat so you don't have any temperature gradient on your flat sample. It is delivered with a small lid you can lay on the top or use as crucible without any border.

3.4. Al Crucible for foils and powders with lid

For foils, powders and other flat samples they could change their shape we recommend our Al crucible for foils and powders. With this crucible you press your sample to the bottom so it couldn't change its position and shape. Not hermetic sealed.

3.5. Al Crucible for small samples with lid

For small granules and samples with small shape we recommend our crucible for small samples with lid. With this crucible your sample is more closely covered by the crucible. Not hermetic sealed.

3.6. Cu Crucible

If you want to measure oxidation effects like oxidation induction time (OIT) or oxidation onset temperature (OOT) it could be helpful to use copper crucibles. These provide a catalytic effect at sample.

4. Other tips and tricks for different samples

4.1. Usage of oil

If a sample has a bad surface it is possible to use silicone oil (e.g. Krytox GPL107) to get a better contact to the crucible. In this case you should take care you don't heat up above the maximum operating temperature of the silicone oil you use. Also it is important to know the Cp values are wrong in this case because you also need to heat up the silicone oil with its specific heat capacity.

4.2. Powdered solids

The sample should be evenly distributed in the bottom of the sample crucible.

4.3. Compact solids

Compact solids, e.g. rubber or thermoplastic, are cut into thin slices with a knife, scalpel or razor blade. A punch tool set can be used to cut disk shapes out of a larger thin sheet. Always size the sample shape to maximize crucible bottom coverage while maintaining flatness. A sample crucible must always be used. Direct application of the sample material increases the danger of contaminating the sensor!

4.4. Films

Discs are punched from films with a hollow drill or punch pliers. The discs should completely cover the bottom of the crucible.

In order to improve the contact between the sample and crucible bottom, the lid should be placed on the crucible, with the convex side down, and sealed.

4.5. Fibers

The fiber can be cut into small pieces, which are then spread parallel on the bottom of the crucible. The fiber is wound around a small rod. The coiled fiber is then removed from the rod and placed in the crucible. A bundle of fibers is wrapped with aluminum foil and cut at both ends. (The weight of the sample can be increased with voluminous fiber materials.) The fiber material with the foil wrapping is then placed in the crucible. The significance of the experimental results can be increased by adding a drop of silicone oil (improves the heat transfer).

4.6. Liquids

Depending on the viscosity, liquid samples can be dropped into the crucible with a thin glass rod, a micro-pipette or a syringe.

4.7. Unstable samples

Unstable samples are tested in special pressure-tight crucibles (optional).

The sensor must be recalibrated when pressure-tight crucibles are used.

4.8. Amount of sample

Depending on your sample you should take care about the weight of your sample.

On the one hand side, it shouldn't be to less, because small thermal effects like glass transitions are hard to detect on the other hand it shouldn't be to voluminous because you will get an temperature gradient on your sample. In this case melting-peaks aren't that sharp. Weight should (depending on e.g. melting enthalpy between 2 mg – 10 mg for metals, and 5 mg – 30 mg for polymers. The sample volume should fit in your crucible. Please be aware gases can be produced if the sample decomposition temperature is reached causing the sample to expand. As this is happening the sample can spill out of the crucible.

4.9. Positioning samples in your crucible

For optimal results you should be sure the sample is laying in the mid of the crucible and the crucible is placed in the mid of the sensor. For powders be sure the bottom is covered with sample completely.

4.10. Cutting samples / Thermal history

Be aware some cutting method can change the properties that you are trying to measure. As an example, a broken polymer (destroyed by hammer e.g.) has a different T_g than the same polymer cut by scalpel. Tearing apart a polymer can cause polymer chains to align and locally crystalize. Always be conscious about how your cutting method might be changing the material. Thermal history might also change the sample properties. Earlier it was suggested heating a second time might improve sample-crucible contact however this only has advantage if the sample properties are thermally stable.

4.11. The right piece

If you cut out an polymer sample from raw materials, your measurement depends on the position of the extraction of the raw material

4.12. Shape of your sample

The shape of your sample affects the measurement. If the shape of your polymer is small the Tg would be higher in comparison to a sample with big shape. In general you must make sure the sample has good contact to the crucible.