GRAPHISTRENGTH® CS 1-25

PROCESSING GUIDELINE FOR DISPERSION into LIQUID EPOXY

Introduction:

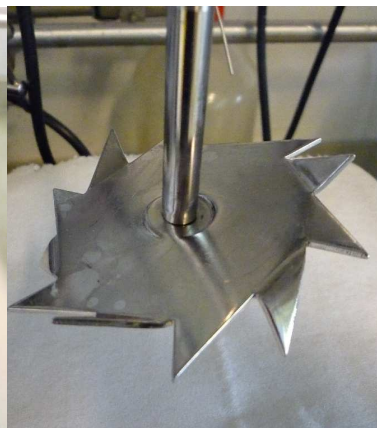
Optimal properties of the final part can be expected with high quality dispersion of the MWCNT.
In order to ensure consistent performances, MWCNT are pre-dispersed in liquid epoxy matrix during the production of the Master-Batch Graphistrength® CS 1-25. Naturally, it is also important to disperse properly Graphistrength® CS 1-25 in the final formulation with typical final loading of MWCNT below 5%. This paper gives information about the key recommended procedures for the dispersion steps of this Master-Batch according to available tools at lab scale.

1 BALL MILLING DILUTION GUIDE

1-1 -Pre-dispersion

- Graphistrength® CS1-25: immerse granules in base resin in an oven overnight at a temperature function of the selection of the base resin: examples 40℃ with epoxy resin LY505 2 or 80℃ for epoxy resin LY556.
- Next day, stir with impeller (picture below) at 500 rpm for 2 hours (to optimize if necessary)
- A fluid liquid with a coarse dispersion of MWCNT is obtained.





1-2 -Ball mill dispersion

Equipment

- Ball mill with:
- Volume of mixing chamber: 250 ml
- Volume feeding hoper: 1000 ml
- Ceramic balls with a diameter of 1.2/1.7 mm

Conditions

- Volume of balls used: 200 ml
- Exit filtering gap: 0.3 mm
- Rotation speed: 4,000 rpm
- Product pushed down the chamber by hydrostatic pressure (0.35 bar)
- Average product's temperature at the exit during milling: 90 ℃
- Number of cycles: 1

Ball milling operations step by step

- Both feeding hoper and milling chamber are pre-heated to 80℃ prior to milling.
- The dispersion is introduced into the feeding hoper and pushed to the milling chamber with a hydrostatic pressure of 0.35 Bar.
- Rotation is initially set at low speed, until the chamber is completely filled, i.e. when the product starts to exit the chamber. It is then smoothly increased to its final value of 4,000 rpm.
- When the product's temperature at the exit starts to increase, the chamber is cooled down in order to stabilize operating conditions. Material can be collected only once temperature and pressure have reached a stable regime. Any material collected under unstable operating conditions is discarded.

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2-ROLL MILLING DILUTION GUIDE

2-1 Pre-dispersion

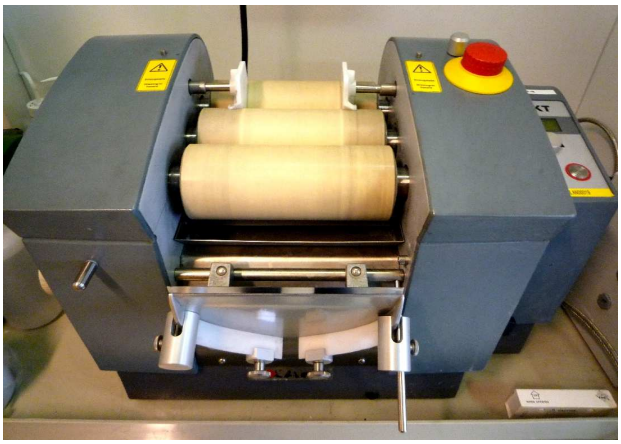
- Graphistrength® CS1-25: immerse granules in base resin in an oven overnight at a temperature function of the selection of the base resin: examples 40℃ with epoxy resin LY5052 or 80℃ for epoxy resin LY556.
- Next day, stir with impeller at 500 rpm for 2 hours (to optimize if necessary).
- A fluid liquid with a coarse dispersion of MWCNT is obtained.

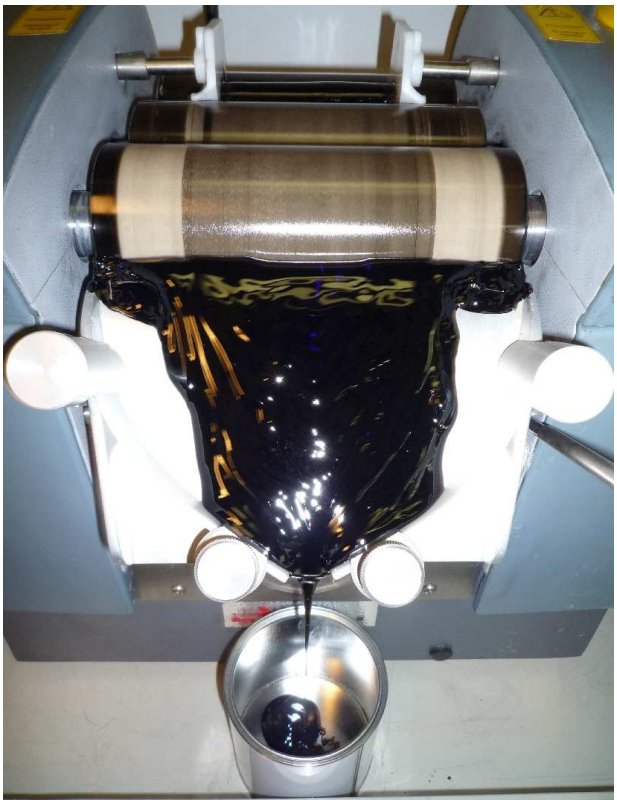
2-2 -Roll mill dispersion

Equipment and conditions

- EXAKT 80E 3-roll mill (electronic calibration recommended)
- First inter-rolls space: 15 microns
- Second inter-rolls space: 5 microns
- Output roll speed: 300 rpm
- Cylinder temperature: ambient
- Number of passes: 1

Output rate is in the range of 2-4 kg/hour.





Pictures of the 3-roll mill with settings and resulting dispersion after milling.

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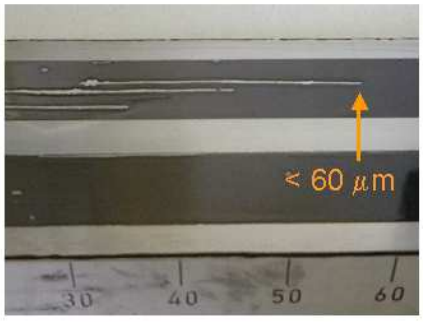
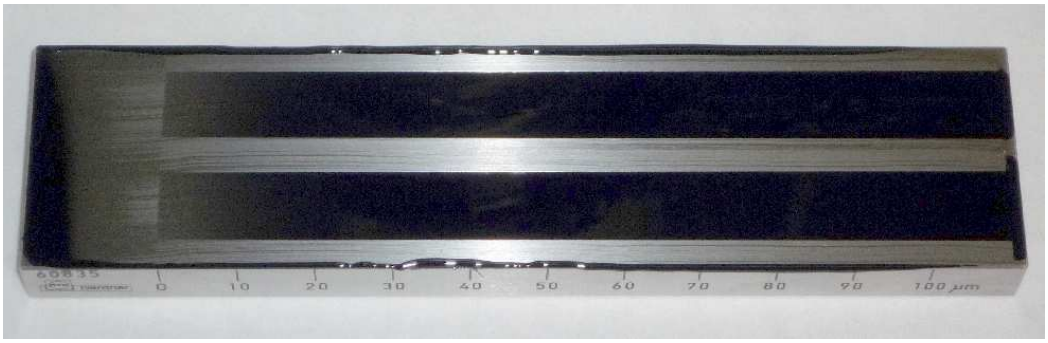
3 DISPERSION CONTROL

- The dispersion quality is evaluated using a North bar. The figure below shows examples of satisfactory vs non-satisfactory dispersions. If dispersion quality is not satisfactory, the product can be placed back in the chamber for an additional cycle.



Similar level of dispersion achievable with the different procedures described above.

Typical value as an example during the control with the North Bar in the case of Roll milling procedure:
This procedure allows obtaining dispersions with aggregates size below 15 microns and even without aggregates as shown on the North gauge below (graduation 0-100 microns).



REMARKS :

Other available tool potentially suitable for the dispersion of the master-batch into a liquid for the step where high shear has to be applied : rotor-stator mixer (e.g. Silverson L4RT at 5000-6000 rpm during 1 to 2 hours. This tool is not recommended due to its limitation to process high viscous liquids.

3 roll milling tool available on the market with possibility to regulate the temperature of the rolls: new options in term of processing

Function of the viscosity of the pre-dispersion produced in the step 1 linked to the viscosity of the liquid epoxy for dispersion and the concentration of MWCNT, 3 roll mill procedure will be preferred.

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