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Training on Cyclic Bending Test Method for Flexible Supercapacitor

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Training Objectives

After this training, the learner shall understand

- The reliability and repeatability of this cyclic bending test method for studying flexible supercapacitor
- How is this cyclic bending test method assessed?
- The bending test system
- How to execute the cyclic bending test of flexible supercapacitor?
- How to calculate the bending radius of the tested supercapacitor?
- How to use this cyclic bending test method to investigate the flexible supercapcitor’s failure?

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1. Assessment of Cyclic Bending Test Method

Experiment

Supercapacitors with 4 types of laminated substrates

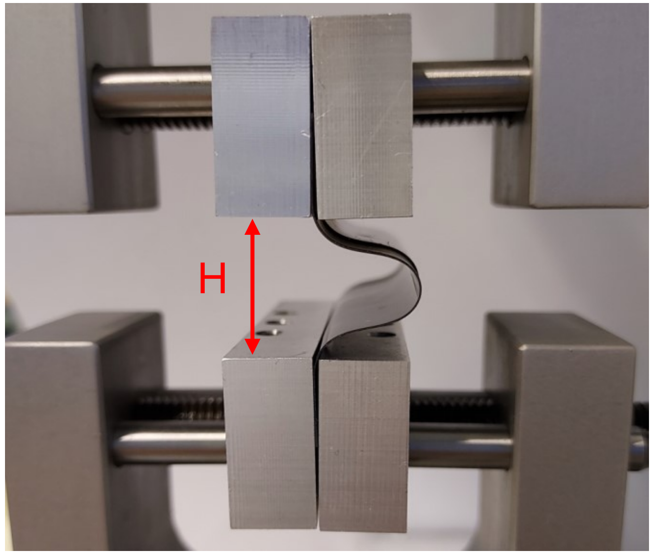
- PLA / Aluminum
- PET / Aluminum
- PLA / Barrier
- PET / Barrier

were tested 3 times when ***H*** = 30, 25, 20, 15, and 10 mm, respectively, by random order.

Measurement System Analysis

To measure the level of variation of the measurement system

Bending Distance (***H***): is the distance between the upper and lower grips of the tester when the sample is bent to the maximum extent (sample is subject to highest impact)



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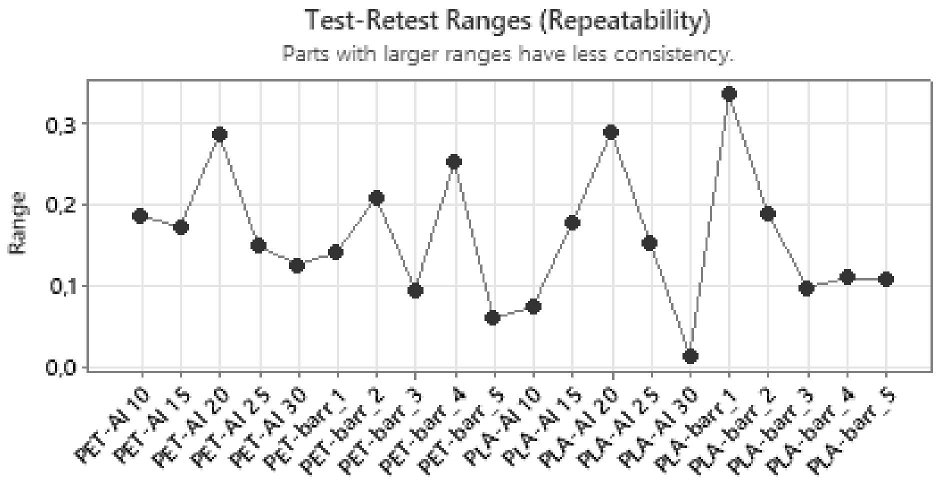
1. Assessment of Cyclic Bending Test Method

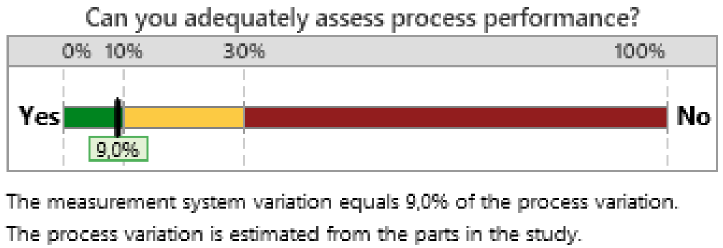
(1) Gage R&R Study for Bending Radius

The bending radii of the bent SC when H reached designed values were measured in all cases.

The results are presented below (e.g. PET-Al-10 means the SC substrate is PET/Al, bending distance H = 10 mm)



Variation in 0.3 mm



High repeatability

General rules to determine the capability of the system:

- < 10%: acceptable
- 10% - 30%: marginal
- > 30%: unacceptable

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1. Assessment of Cyclic Bending Test Method

(2) Variation in Bending Radius

For 20 cases (4 types of substrates, 5 bending distance for each SC type) of test, we calculated bending radius variation level (***Δr***), and average bending radius (***r***).

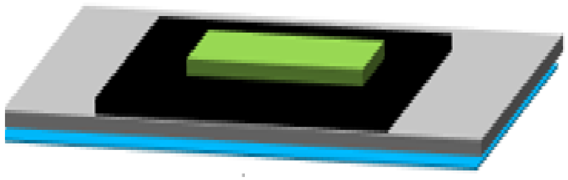
Then, we computed ratio = ***Δr*** / ***r***. Results:

- All cases: < 8%
- 17/20 cases: < 5%

Low level of variation

Sources of variation

- Uneven thickness distribution across the sample (main)
- Operation
- Imaging
- Others



If the SC architecture can be redesigned in a way to minimize the thickness difference across the SC, the variation of bending radius can be lowered!

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1. Assessment of Cyclic Bending Test Method

(3) Stable SC Structure in Cyclic Bending

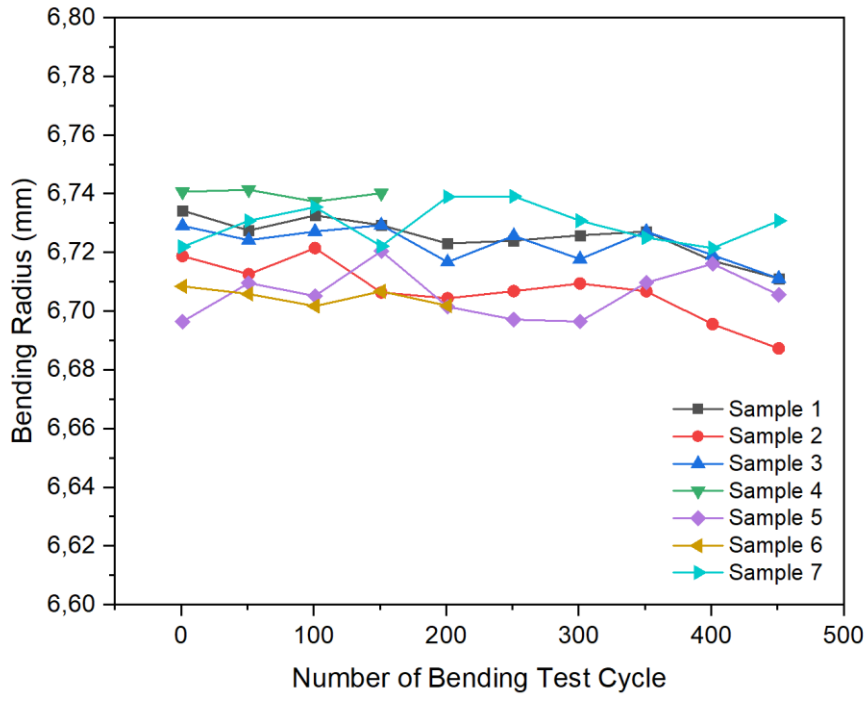
To investigate how stable the SC structure is during the cyclic bending, we measured the bending radii of SC when it was bent under the designed bending distance (H) in the first cycle of every test.

The test was conducted in 50 cycles intervales, followed with electrical measurement.

Thus, we got the bending radii of SC at cycle 1, 51, 101, ... until 451 (maximum 500 cycles test)

And calculated the variation of calculated bending radius.

Example: 7 printed flexible supercapacitors with PLA/barrier substrate in 500 cycles bending test.



Variation in 0.03 mm!!

This shows that the bending radius of the sample is highly stable during the cyclic bending test.

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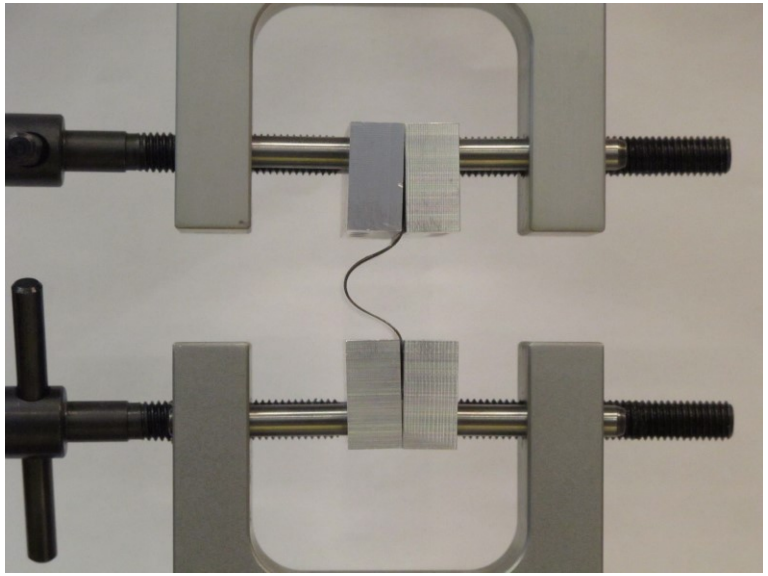
1. Assessment of Cyclic Bending Test Method

Summary

Research: assessed a cyclic bending test method for printed flexible supercapacitor (SC).

- 4 material stacks (substrates)
- 5 different bending radii (distance)



Conclusion

- Variation of measurement system is **acceptable**.
- Test method is **repeatable**.
- Flexible SC's bending radius is **stable** during cyclic bending

This cyclic bending test method is verified to be repeatable and reliable for evaluating printed flexible SC!

However, for specific device, the bending radius needs to be specified, since for different materials, even under the same H value, the bending radii are different.

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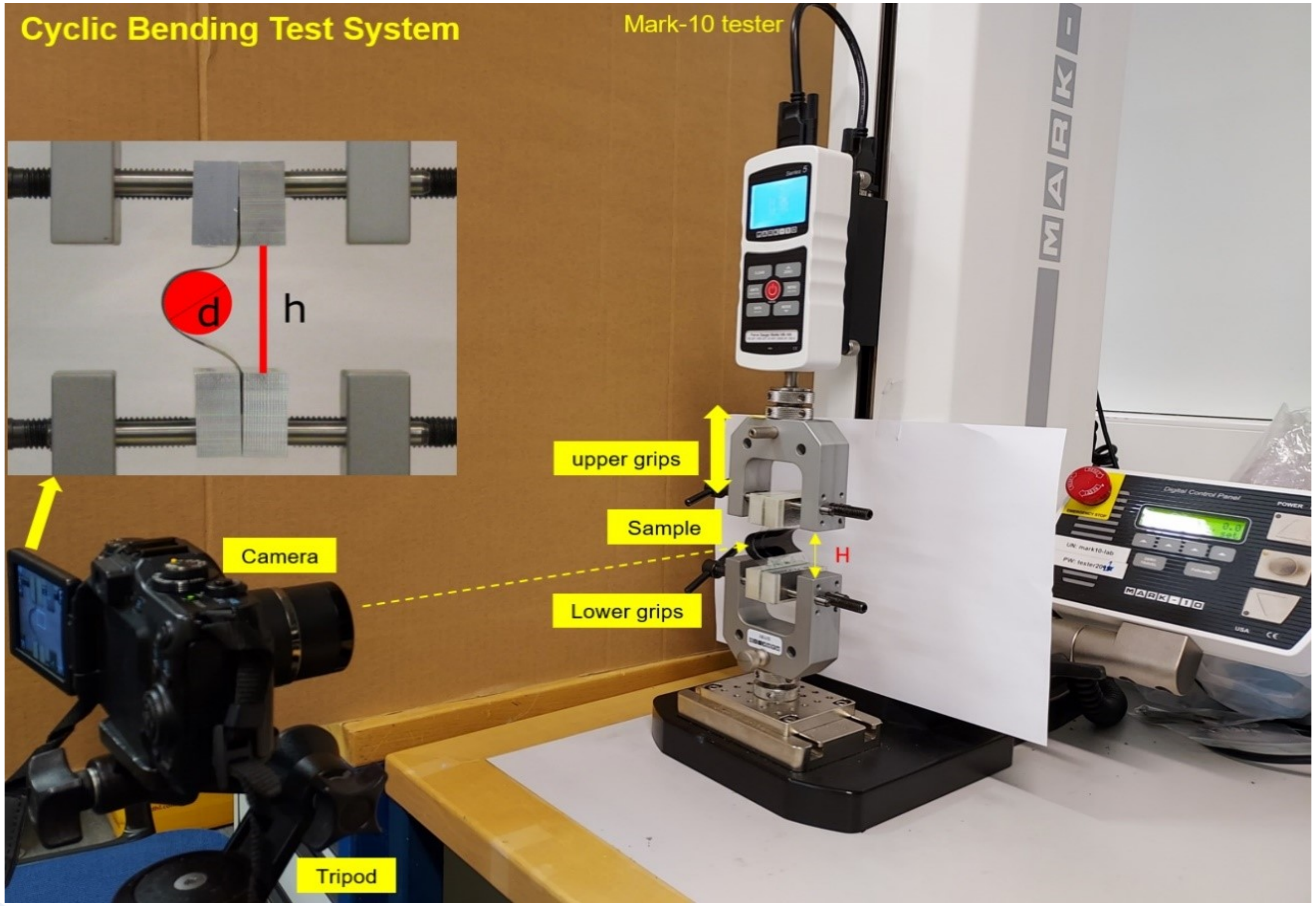
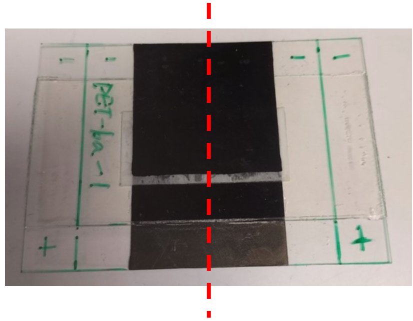
2. Cyclic Bending Test Setup/System



Equipment

- An ESM 303 Mark-10 motorized tension test stand
- An optical camera (e.g. we used Canon G11 camera)
- A tripod
- A level ruler
- Image processing software (Inkscape)



Bending axis

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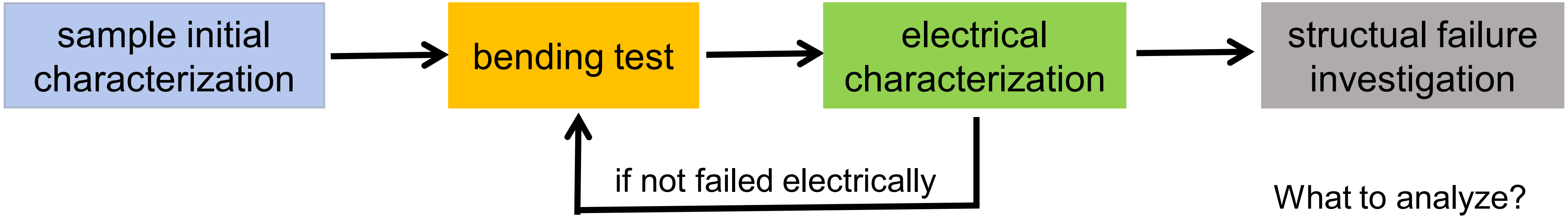
3. Application of Bending Test Method for Supercapacitor

Now, suppose you have some flexible supercapacitor samples, or you plan to use do cyclic bending study to flexible supercapacitor,

how to use this cyclic bending test method to test the samples?

3. Application of Bending Test Method for Supercapacitor

General steps of failure investigation of supercapacitor and involved issues



sample initial characterization

What to characterize?

e.g.
Mass
Electrical properties

bending test

Test parameters;

Operation;

Bending radius calculation;

Number of samples

electrical characterization

Electrical properties to characterize;

Electrical failure criteria;

Device for characterization;

Time duration for characterization

structual failure investigation

What to analyze?

Procedure of analysis

Devices for analysis
- optical microscope for surface check
- non-destructive analysis
- SEM analysis
- ...

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3. Application of Bending Test Method for Supercapacitor

Step 1: To identify the Bending Distance (H) values

Why Bending Distance?

for such cyclic bending test, two approaches can be considered:

(1) To identify bending distance: e.g. to design the test that the SC samples are to be bent until H = 20 mm, whereas the bending radius need to be measured/calculated.

If the samples are fabricated using the same materials, architecture, dimensions, the bending radii of different samples are expected to be very similar.

(2)To identify bending radius: e.g. to design the test that the SC samples are to be bent until r = 3.5 mm, whereas the bending distance that lead to 3.5 mm bending radius needs investigation.

Method (2) is much more complicated, it needs more trials to identify the H value to lead to the ideal r. And for testing samples with different materials and/or structure, the H values are different for

reaching the same r value. Thus, method (1) was selected.

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3. Application of Bending Test Method for Supercapacitor

Step 1: To identify the Bending Distance (H) values

(1) Do some trial bending using the SC sample, to identify the highest and lowest H values.

- How to identify highest H: The up point bending to which can causes considerable impact to the active area of the SC can be considered as point for highest H

meaning if the SC is bent to even lower extent (i.e. bent to higher H value), it causes very light impact to the SC, then, it's not necessary to bent to lower extent.

- How to identify lowest H: The low point being to which can cause very severe impact to the active area of SC can be considered as point for lowest H.

Meaning, if the SC is bent to even lower H value, it does not cause more severe impact to the SC's active area, or it causes too rapid failure.

(2) define all H values for tests: e.g. if you have defined highest H = 30 mm, lowest H = 10 mm, you may choose to define 5 mm gap among different H values for tests - 30, 25, 20, 15, 10 mm.

Using too few H values makes it difficult to get detailed understanding;

Using too many H values would make it time-consuming.

3. Application of Bending Test Method for Supercapacitor

Step 2: Define test parameters

(1) **Test speed**: If the speed is too high, the failure can occur so soon that it is difficult to investigate the failure initiation and evolution. If the speed is too low, the test would be time-consuming.

1) To define it, relevant test standard(s), published papers of similar test can be referred to.

2) Do some trial tests to the lowest H values (highest impact to SC) with the potential speeds, and consider the duration of test until failure. To determine the speed based on it.

One can calculate the duration of test in this way:

One cycle of test: sample is bent from straight status to the programmed H value, and return to the start point (sample is straight).

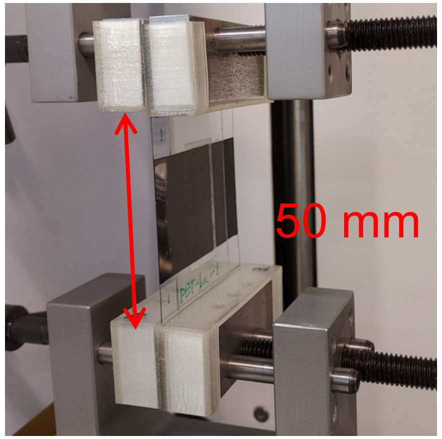
e.g. If the distance between grips is 50 mm at beginning, H = 20 mm,

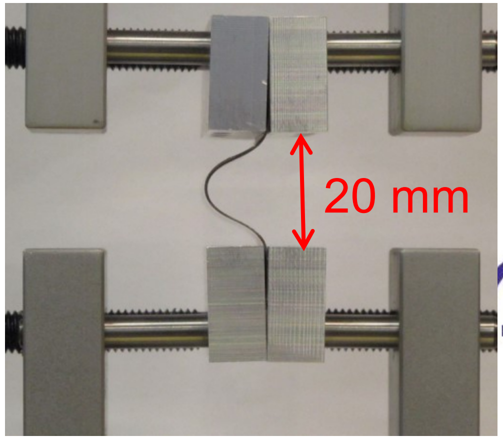
one cycle travel distance is (50 - 20) x 2 = 60 mm.

If test speed is 300 mm/min.

one test of 50 cycles takes:

60 mm x 50 / 300 = 10 min.





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3. Application of Bending Test Method for Supercapacitor

Step 2: Define test parameters

(2) **Number of test cycles** for each time test: it is not aimed at testing until failure in one test. To enable investigation of failure initiation and evolution, it is better to test with certain number of cycles of intervals, e.g. test 50 cycles every time, followed with electrical measurement to check if it is failed.

1) Try to test few samples with 100 cycles, 300 cycles, 500 cycles, ... and **define the estimated number of cycles where failure is most lilely to occur**.

2) Define the test interval (number of test cycle for each test): E.g. if you find most samples failed completely (or severely) at 400-500 cycles, 50 cycles for one test ideal is suitable. If most samples failed completely at 800-1000 cycles, 100 cycles for one test is ideal. It's a tradeoff of duration of test and convenience of investigating failure evolution.

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3. Application of Bending Test Method for Supercapacitor

Step 3: Define electrical characterization method, device, failure criteria

Depending on the interest of research, decide the electrical measurement of tested SCs.

(e.g. in our research of SC, we used Marcoo device to measure the capacitance, ESR, and leakage current of tested SCs to characterize the performance of SCs. Our Marcoo device enables to measure at most 8 samples simultaneously).

Consider how can measure easily and accurately.

Define the electrical failure criteria:

e.g. in our previous research, we identified industrial criteria (based on some standards and published papers of similar study) for capacitance, ESR, leakage current, respectively.

When one of the failure criteria is reached, the SC sample is considered to be failed, which would terminate its further test.

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3. Application of Bending Test Method for Supercapacitor

Step 4: Plan the characterization (procedure) of structural change/failure.

1) For some tested SCs, there maybe visible failure e.g. cracking, material peel off, electrode flaking off, etc. Optical microscope is ideal to check the overall condition of the samples without breaking the sample.

Passible outcome: (1) no physical or structural change on the sample's surface.

(2) crack and/or material lost in some areas of the sample's surface. Based on this, one may get initial idea where the failure may occur.

2) Non-destructive investigation of internal structural condition/failure: e.g. X-ray micro-CT or other equipment that enable to observe the sample's internal condition without breaking the sample.

Outcome: idetify the location and form/sign of physical failure.

3) Select interested areas in interested samples for more detailed analysis using e.g. SEM

Outcome: identify the failure mechanism.

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3. Application of Bending Test Method for Supercapacitor

Step 5: Plan the number of samples

From the statistical point of view, usually at least 5 samples of the same type are needed for the same test. Larger number of samples can enhance accuracy of data, but consume more time of research.

Do not forget reference samples.

Based on test parameters (test speed, Bending Distance values) and electrical measurement, the time for research can be estimated.

- *Note: for supercapacitor, usually the electrical measurement can take much longer time than the bending test. Considering how to organize the electrical measurement efficiently can save time.*

Step 6: Start bending test

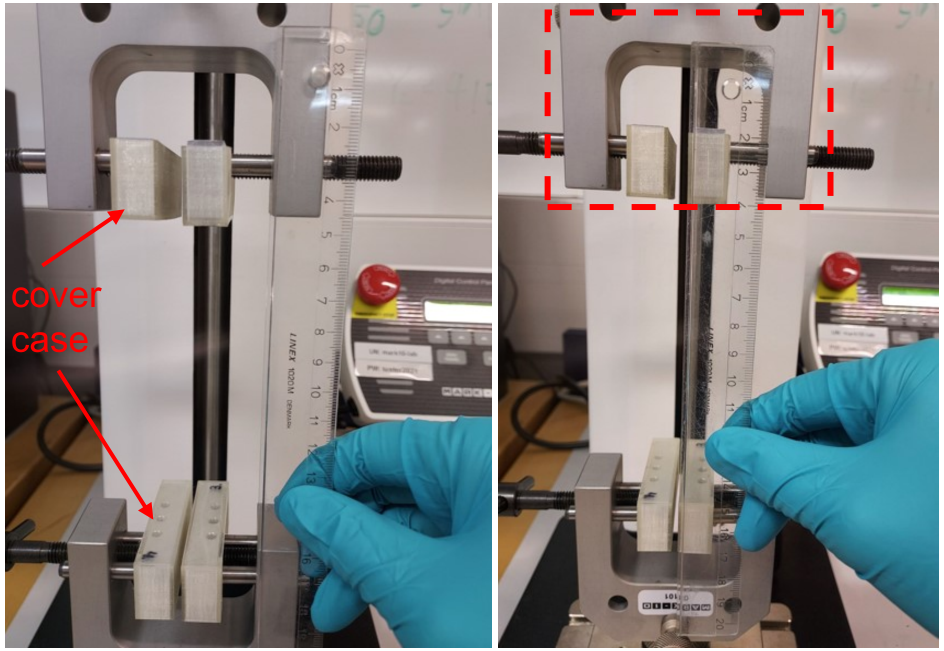
- the detailed procedure for bending test is introduced in section 4.

4. Test and Measurement Procedure

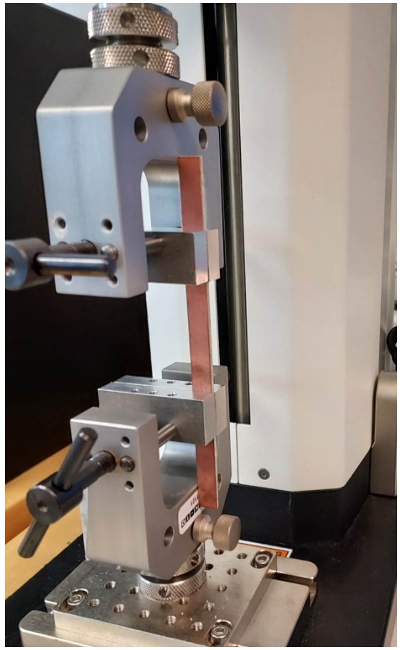
(1) Check the tester

- Install the polymer cover case onto the steel fixture, to protect sample from being damaged by sharp corner when fixed.
- Check and ensure that the upper and lower grips are aligned, so that sample can fixed straightly later on.

can be rotated



Check alignment of tester and grips / fixtures



To check and ensue that the upper and lower clamps are aligned vertically.

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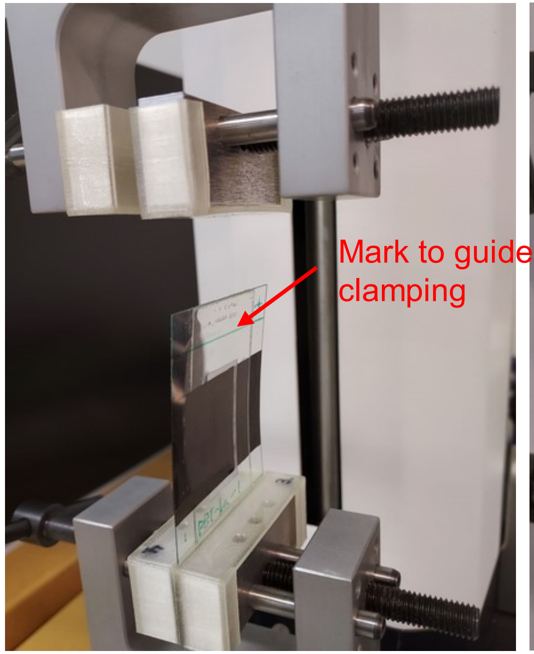
3. Test and Measurement Procedure

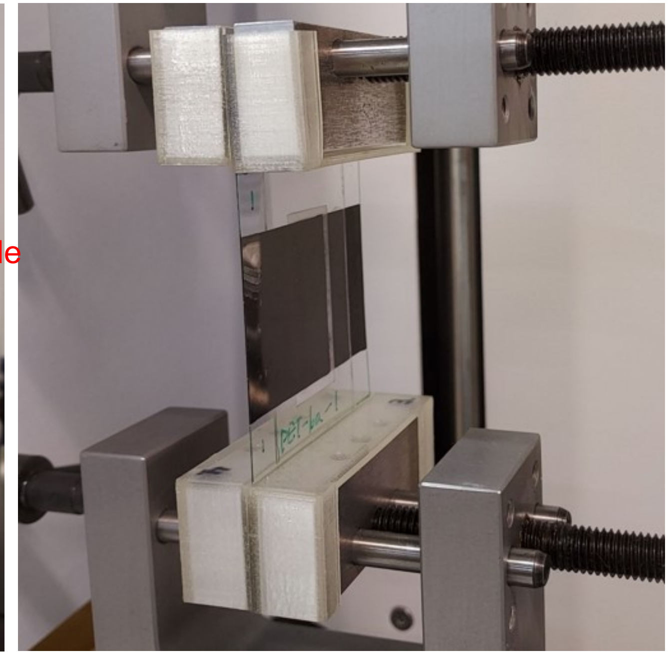
(2) Fix a SC Sample

- Fix the lower end of the SC first – Fig. (A).
- Move the upper fixture by controling the control panel. Move to the point slightly lower than the marked clampping line (this is critical for next step).
- Close the upper clamps, the sample is always bent a bit, then, move the upper grip upwards slightly to ensure the sample is straightly but not subject to high tension. (Fig. B) The tension is shown by the value on the screen. (Fig.C)

(note: do not expect it to be 0, since when clap is closed, it moves upwards slightly, causing some tension, ~ 20 N tension is acceptable)



(A)



(B)

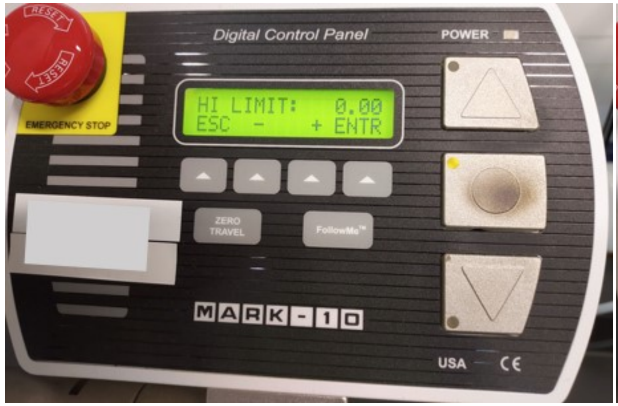
(C)

3. Test and Measurement Procedure

(3) Set the test parameters

- **Zero point**: once the sample is fixed straightly without significant tension, this position can be set as zero point by pressing ZERO TRAVEL button on Digital Control Panel (Fig. A)
- **High and Low Limit**: set the High Limit to 0 (Fig. B), Low Limit to the desired bending distance – If you expect the sample to be bent for maximum 20 mm, then, Low Limit is set to – 20. (Fig. C)
- **Number of bending test cycle**: set based on your test plan. If you wish to test with 50 cycles interval, then, set it to 50 (Fig. D).

(A)



(B)

(C)

(D)

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3. Test and Measurement Procedure

(3) Set the test parameters

- **Test speed**: Trial and reference are neded for determining the test speed. Too fast test can lead to failure too soon that the failure evolution and mechanism cannot be tracked or studied well. Too slow test consumes test time. Relevant test standards can also be applied.

e.g. In our research, many references used 300 and 400 mm/min for cyclic bending test. By trying speeds of 300, 350, 400 mm/min, we selected 350 mm/min for the test. (Fig. E)

Save setting: after each change of setting, press Save Change (Fig. F), until you see SAVING CHANGES (Fig. G)

(E)

(F)

(G)

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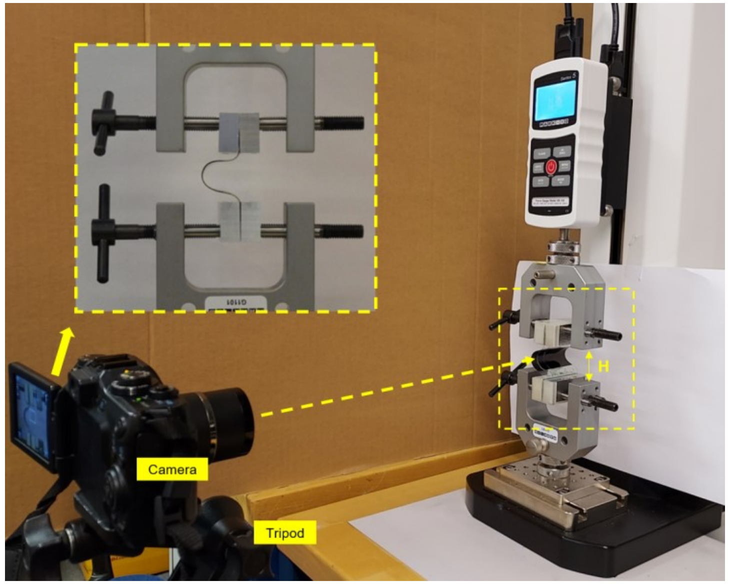
3. Test and Measurement Procedure

(4) Imaging the bent SC

1) Bend the SC by moving upper grip downwards, and manually stop the moving when it moved to the lowest point (when the value of position shows – 20.00, if the Low Limit was set to – 20).

2) Place a tripod in front of the sample, use a level ruler to measure the flatness of tripod's surface (Fig. A), and adjust tripod until it is aligned with horizontal line (its surface is not tilted).

3) **Adjust camera and imaging**: fix the camera on the tripod, adjust the tripod height until the camera view focus is on the middle of the bent sample. Adjust the distance between camera and sample until the view of the sample is clear and suitable in the image.



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3. Test and Measurement Procedure

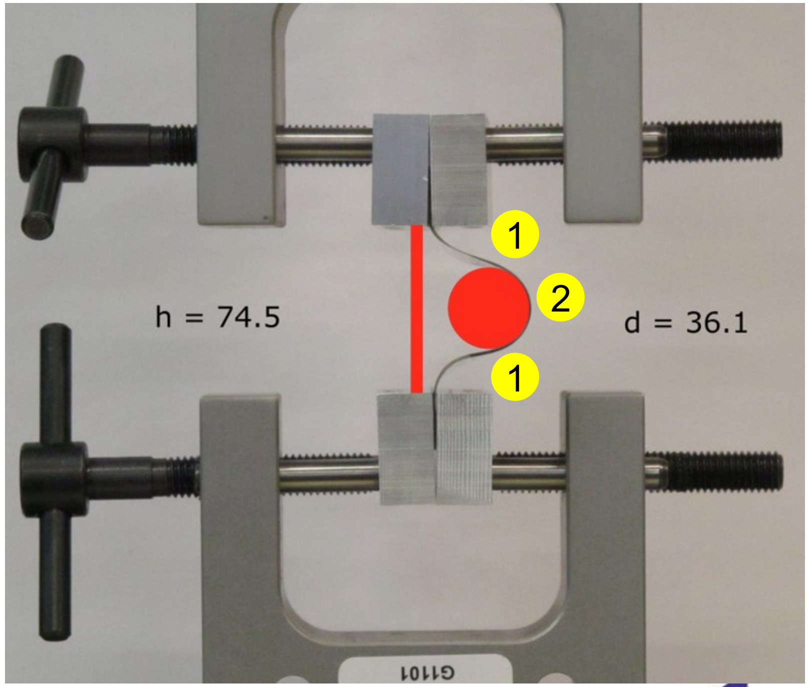
(4) Calculation of Bending Radius

1) Insert the image to image-processing software, e.g. Inkscape. Fit a circle into the empty area of the bent sample as much as it can. Insert a rectangle to fit the upper and lower grips.
2) Read the values of the circle's diameter ***d*** and the length ***h*** of the rectangle from the image software.
3) Calculation: the ratio of ***d*** and ***h*** in image equals their ratio (***2r*** / ***H)***) in the real tester. Thus:

$$\frac{d}{h} = \frac{2r}{H} \quad \Rightarrow \quad r = \frac{dH}{2h}$$

Experience – criteria for good image
point 1 – you can almost not be able to see the side downwards of upper grip AND not see the upperside of the lower grip.
Point 2 – you should see the bent sample almost only a line, meaning not seeing much of its inside or outside surface.

However, this is very idea case. In practice, it's hardly to get such since the clampping of the sample causes slight deformation of the flexible sample.



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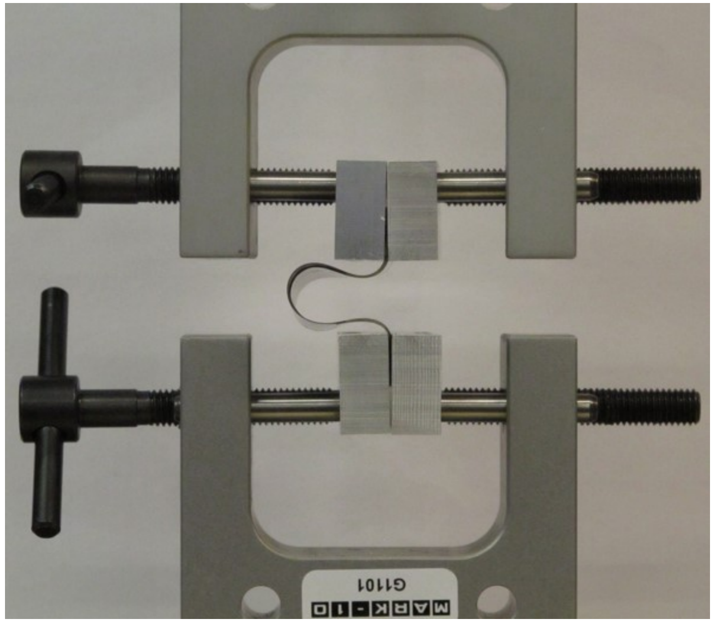
3. Test and Measurement Procedure

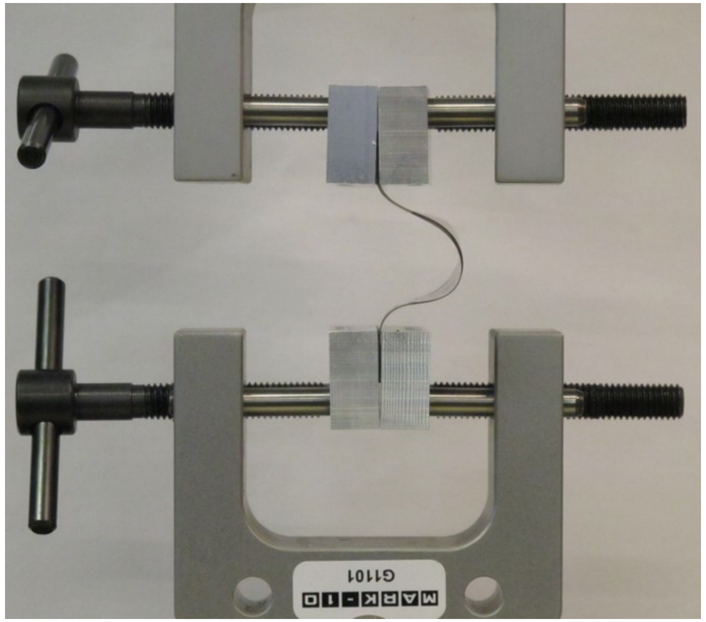
Examples of poorer (and more possible) images

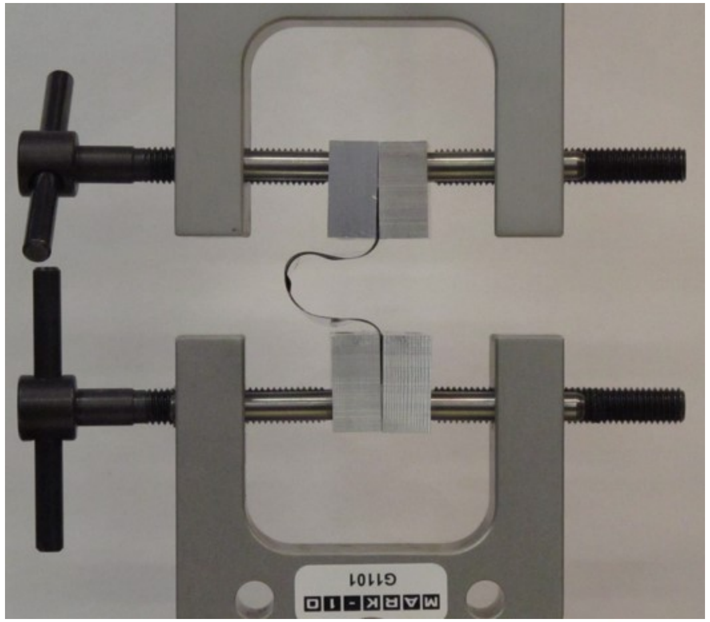
When these happen, what to do? Or how to avoid?

- Check if the sample is fixed straightly, and not tilted.
- Check if the initial tension in the sample is too high.

Sample’s quality (components alignment in assembly) can also cause such problem.







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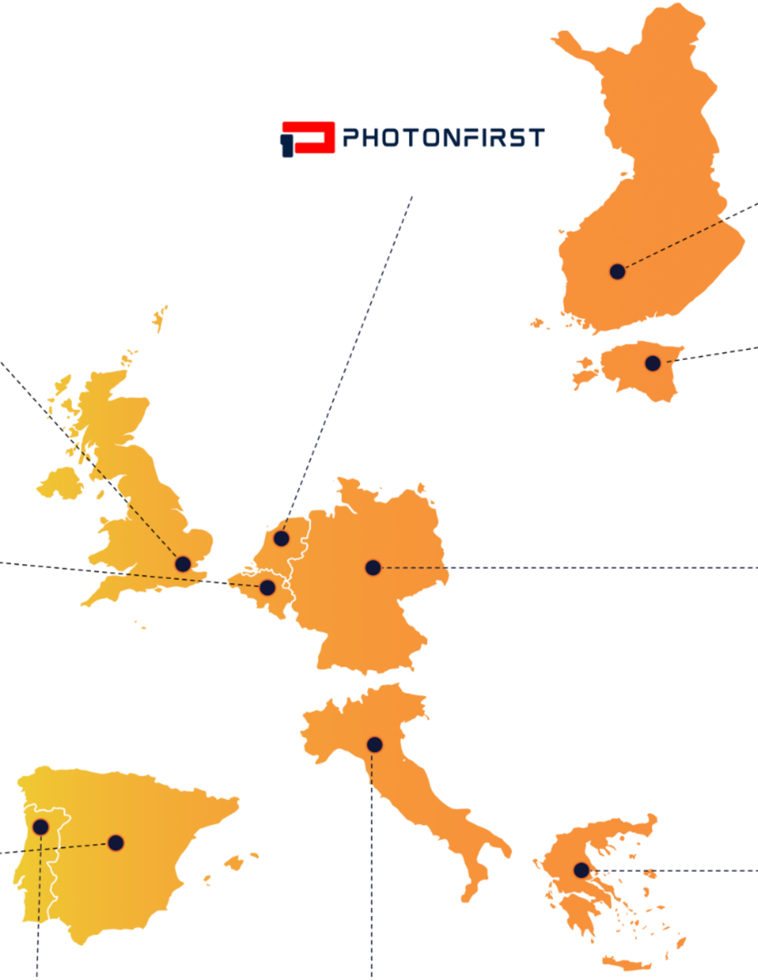

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Thank you

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for Flexible Supercapacitor

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