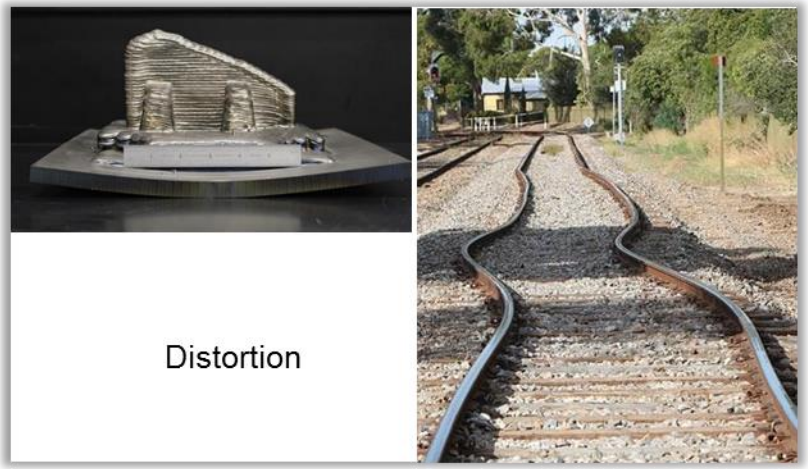
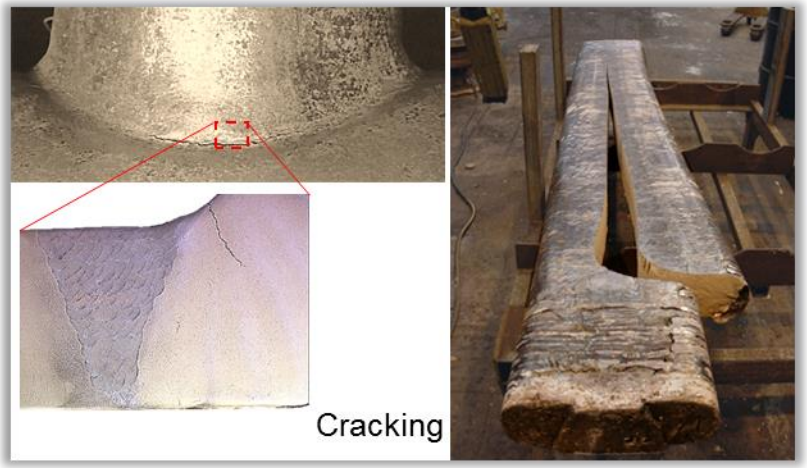
Depth profiling with XRD

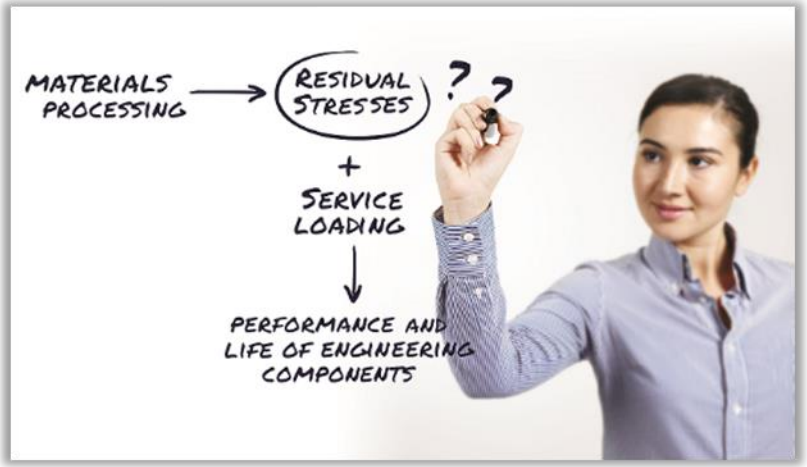
Dr Jeferson Araujo de Oliveira

Managing director at StressMap, The Open University

BSSM XRD Workshop and Exhibition







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Introduction

Why to measure stresses through the depth?

- It is critical for accurate structural integrity assessments

- Essential for accurate fatigue life and stress-corrosion-cracking estimations.

- An important parameter to compare and optimise the benefits of engineered residual stresses in safety-critical/high-performance applications

- Critical for predicting distortions upon material removal

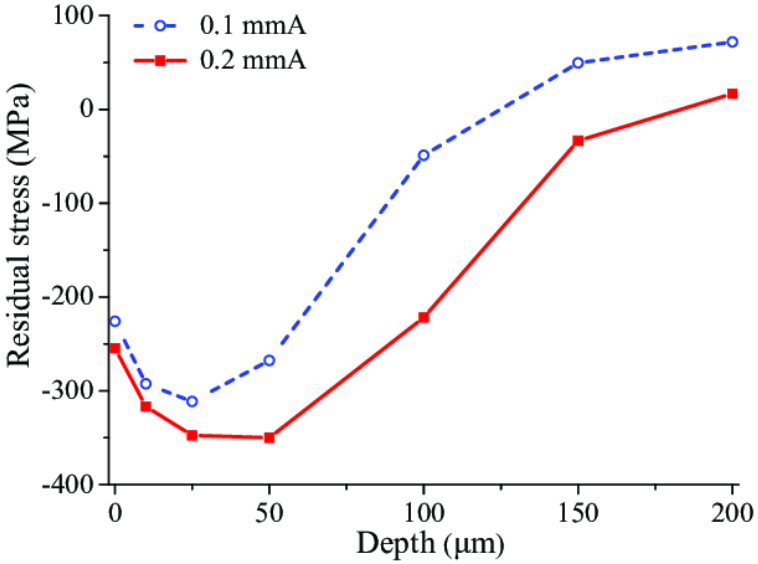


Image: DOI: 10.3390/ma12050743

Layer removal in theory

Depth profiling involves the removal of material

Stresses at deeper layers redistribute

A correction procedure was published in 1956[1]

Assumptions for this correction procedure:

- Layers are removed from the entire surface
- Specific geometries: Flat plate, cylinder or tube
- Material removal does not introduce additional stresses
- Stress redistribution is elastic

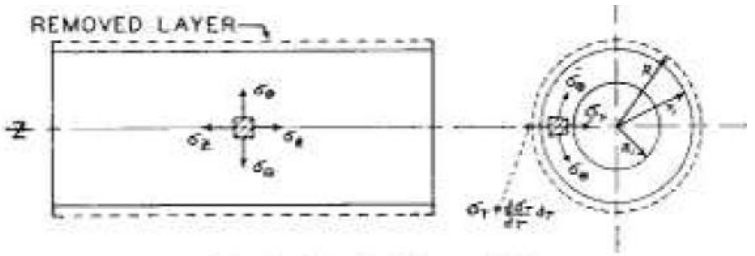


Fig. 3—Case 3, hollow cylinder

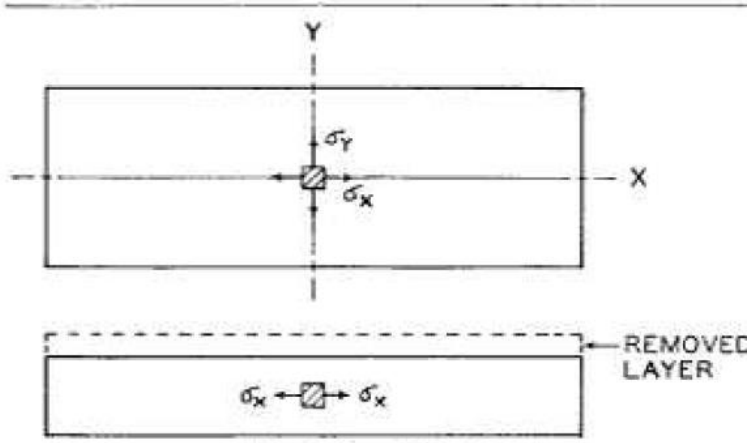


Fig. 4—Case 4, flat plate

[1] M. G. Moore and W. P. Evans, *SAE Technical Papers*. 1958, doi: 10.4271/580035.

Theory

Here's an example of the effect of the correction on the results [1].

Note that errors become more significant with depth and stress magnitudes, in this example reaching about 15ksi (~100MPa) at 0.008 in (0.2mm) depth.

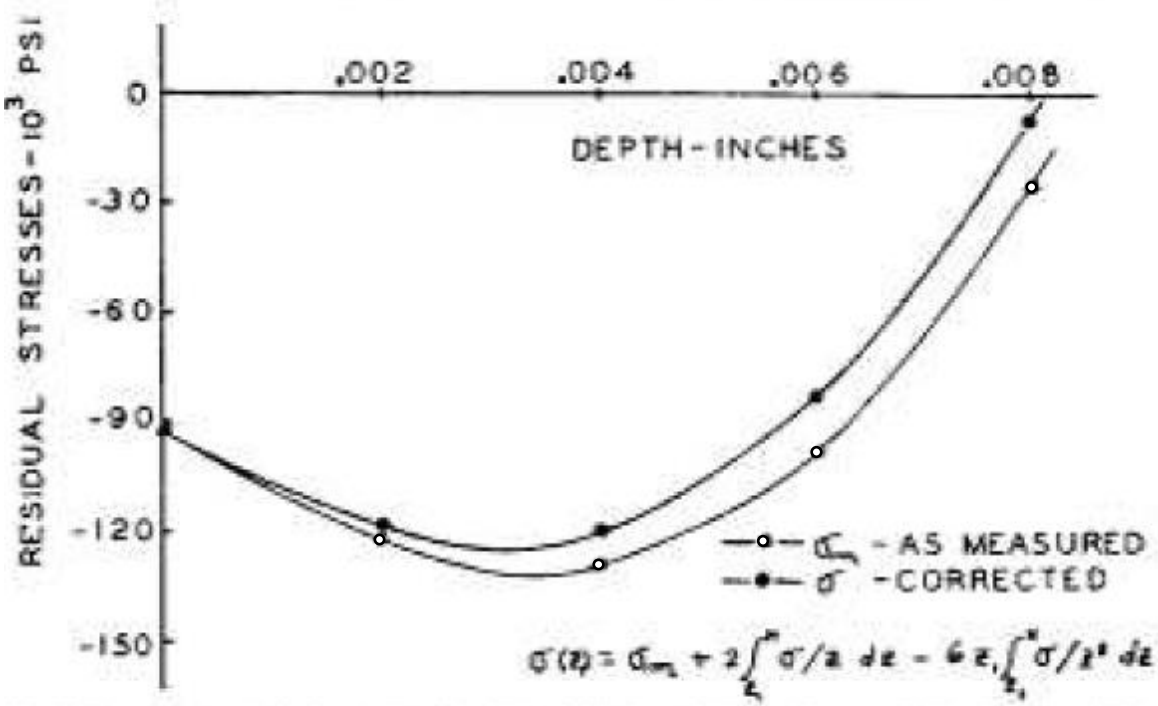


Fig. 13—Flat plate, Case 4, shotpeened leaf spring, SAE 5160 steel (0.192 × 1½ × 12 in.)

[1] M. G. Moore and W. P. Evans, *SAE Technical Papers*. 1958, doi: 10.4271/580035.

Practice

In practice, we have additional challenges:

- We normally cannot remove a layer from the entire surface of the specimen
- Electropolishing is the most popular material removal technique for metals

  Pros:
  - It does not introduce additional stresses
  - Depth is somewhat controllable
  - Small increments achievable

  Cons:
  - Limited material removal rate
  - Depth steps are inaccurate (~±5 µm)
  - In-plane positioning is inaccurate
  - The patch is not necessarily flat and often presents stepped edges

So how can we get reliable results?

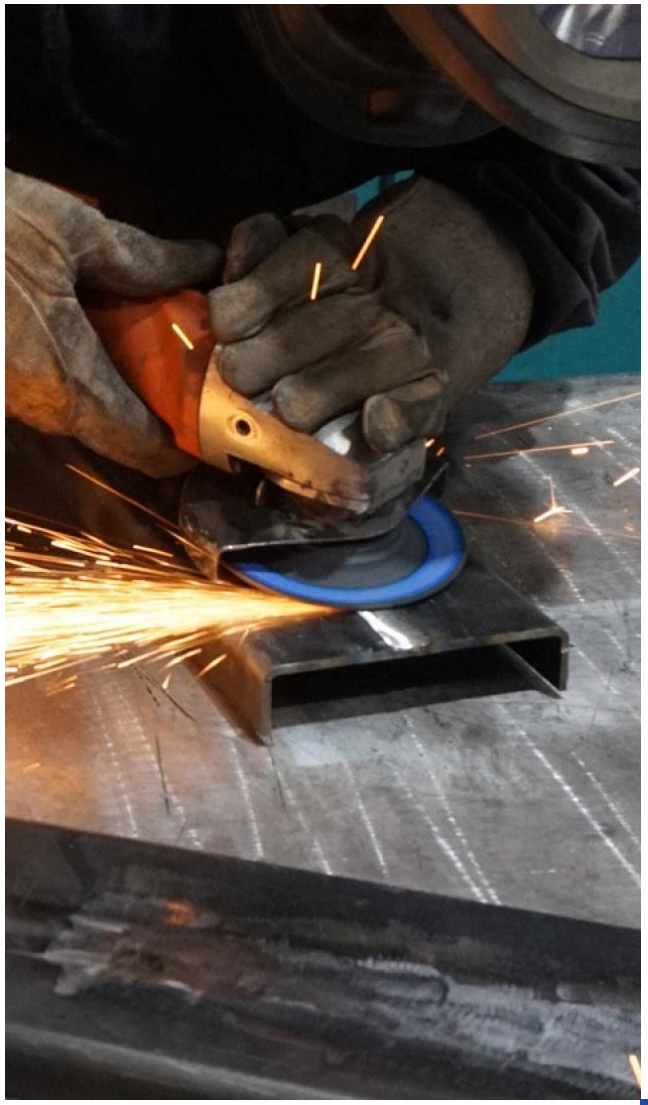
Practice – Material removal rate

How to overcome the limited material removal rate from electropolishing:

- Light grinding then electropolishing
  - low depths (~1-2 mm)
- EDM machining then electropolishing
  - Deeper than 2 mm.

Finally, electropolish least 100 µm to remove any residual stresses induced by grinding or machining.

Do not do this! =>



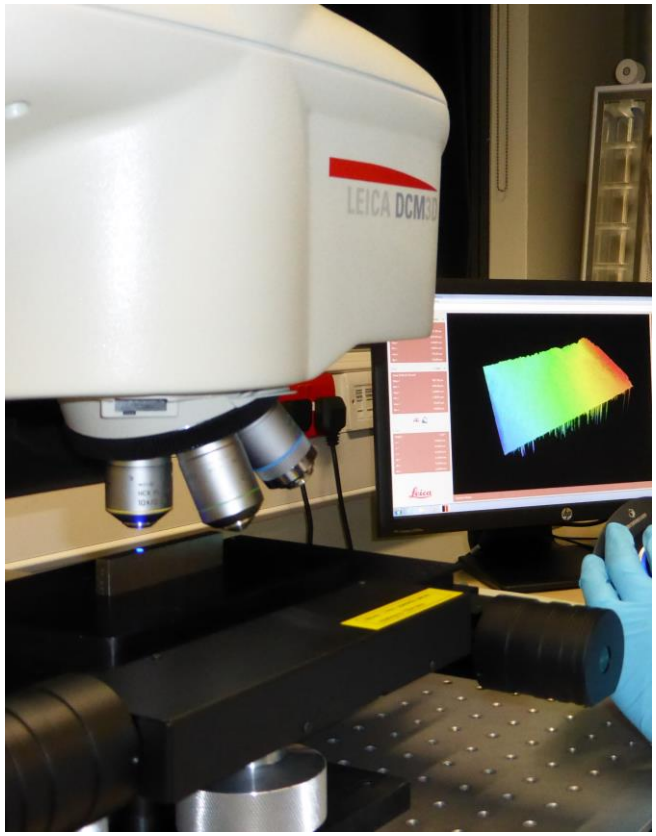
Practice – Depth inaccuracy

How we deal with the depth inaccuracy

We measure the depth increments and report them, instead of the nominal figures.

Here's how:

- Measure the specimen's surface (reference)
- Define reference areas, not electropolished
- Masking is sometimes essential
- Alignment must be repeatable to microns
  - So specimen is removed, reset and measured again to ensure repeatability
- Depth is measured after each electropolishing step
- When close enough to nominal target, record and report the real depth.



Practice – Example

How to deal with the positioning inaccuracy?

Here is a common example:

You are tasked with 42 measurements on a 15 by 15 mm grid in a heavy specimen (>100kg).

You can choose between two different electropolishing machines:

- Bench-top machine that can electropolish up to a 30 x 10 mm^2 patch in one go.

- A portable system, that can electropolish ø 6mm diameter patches

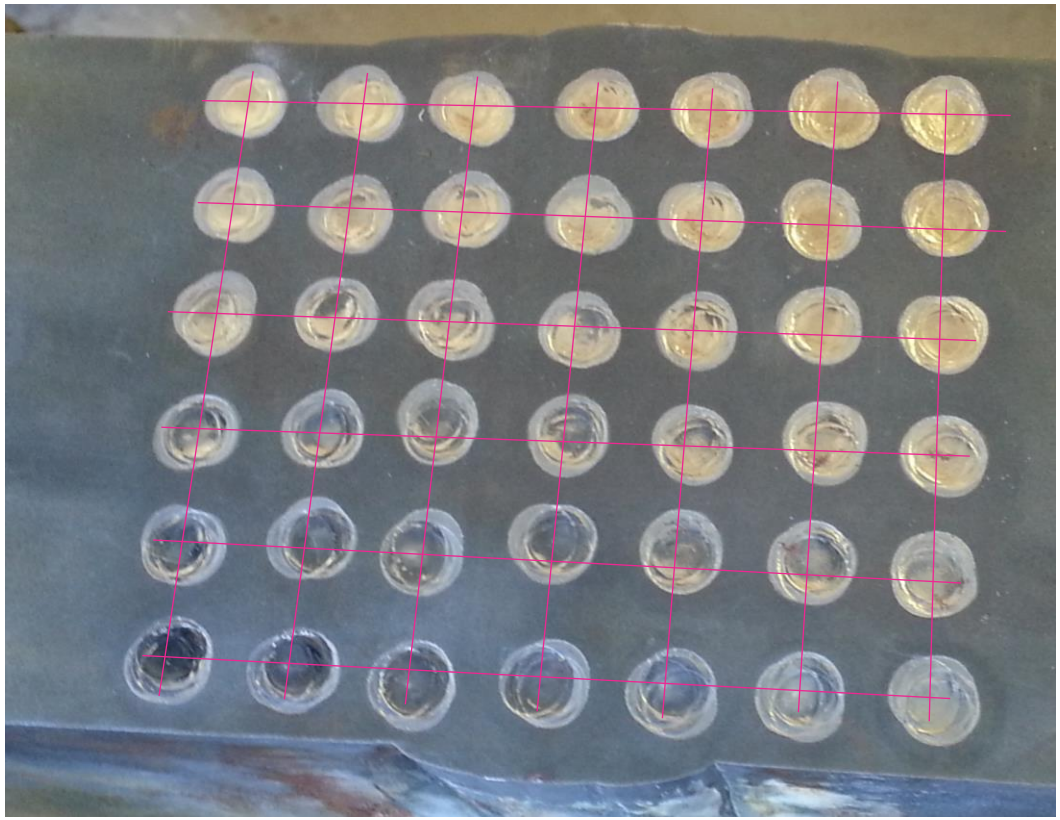
Which one do you choose?

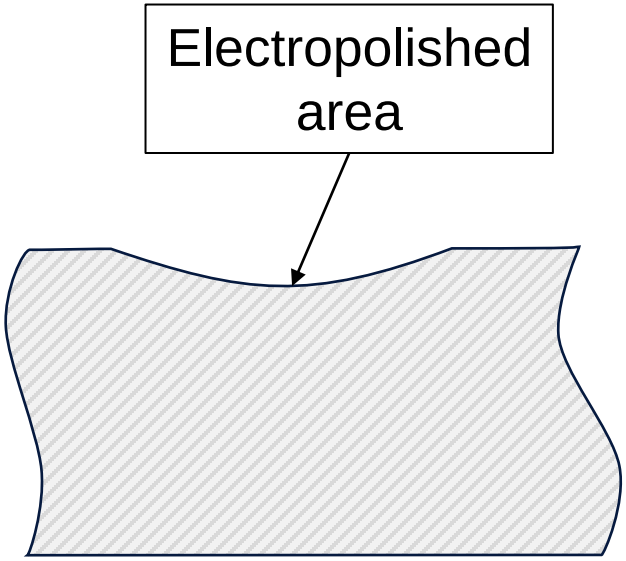
The portable one, because:

- The overlap between electropolishing patches create steps, and we are measuring a grid
- The specimen is too heavy to put on top of the machine as well!

Practice - Example

Okay, so you mark the specimen, perform the electropolishing and it looks like this:





We cannot call this a regular grid, can we?

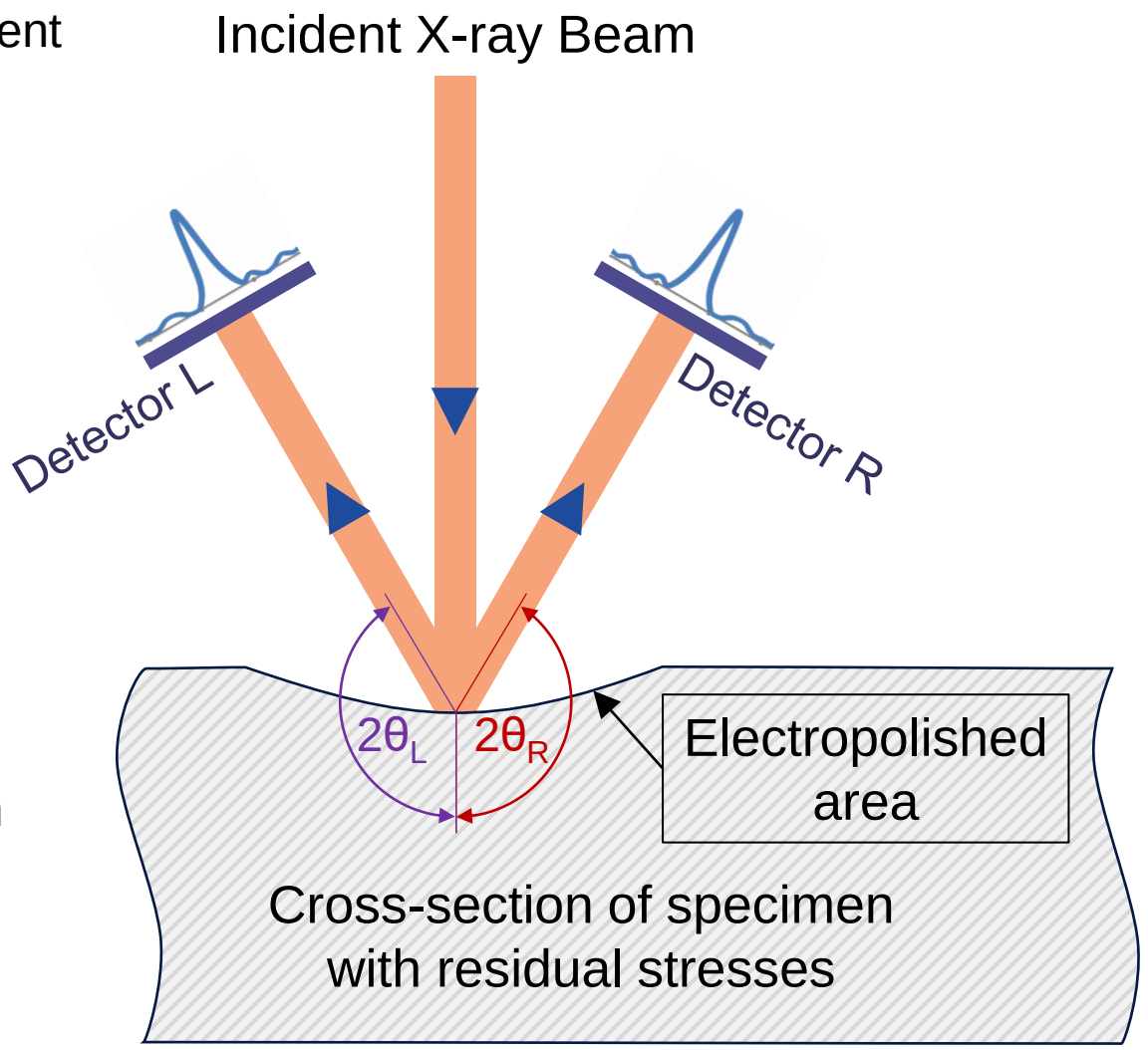
Practice - Example

This is how a good measurement should look like:

How would you ensure this is how the measurement is set up?

Positioning each measurement individually, accounting for the deviation from the nominal grid.

Then report the actual location of each measurement point.



Common problems

Misaligned electropolished spot

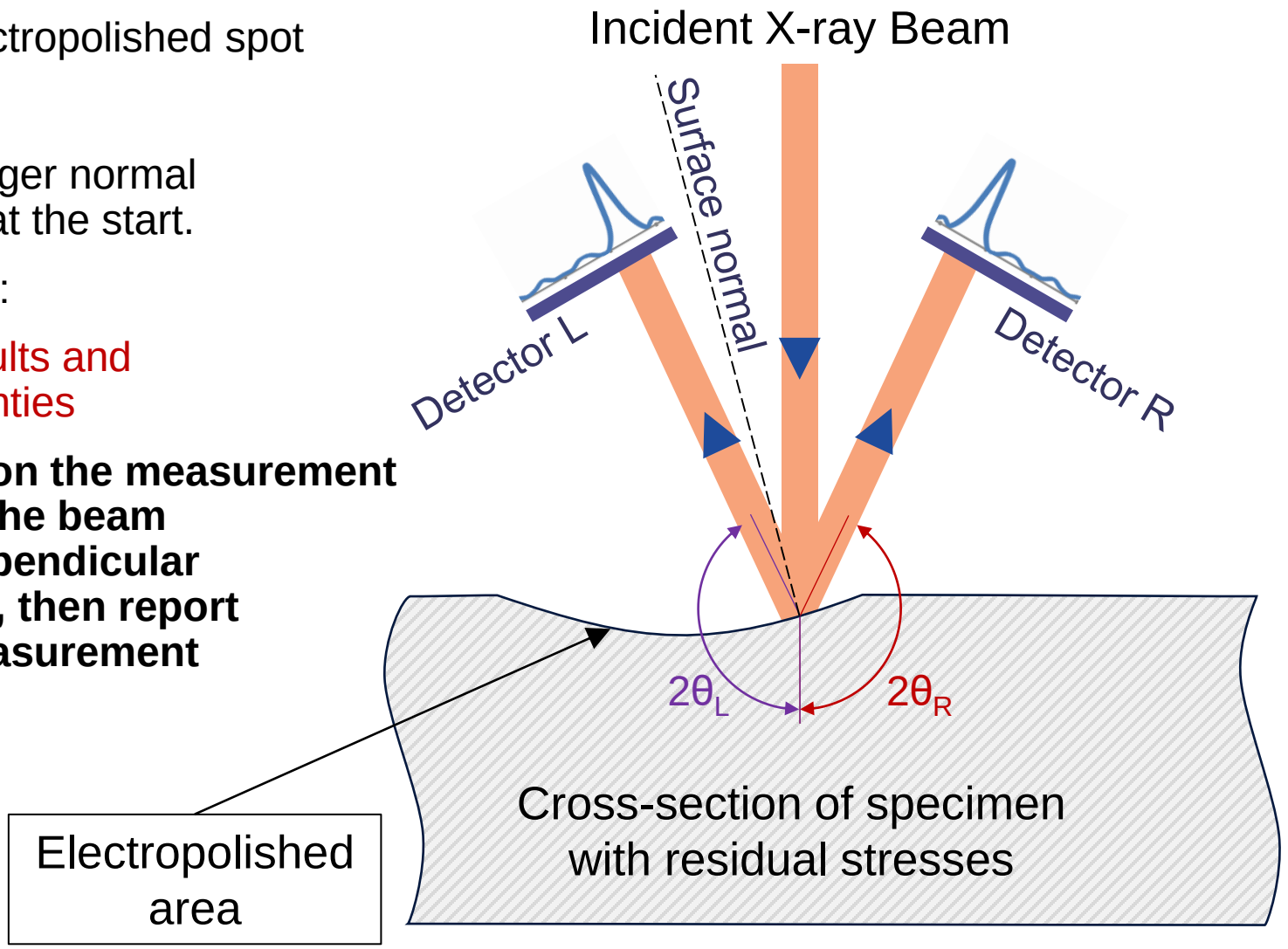
Problem:

Beam is no longer normal to the surface at the start.

Consequences:

Bias in the results and larger uncertainties

Always position the measurement point so that the beam is initially perpendicular to the surface, then report the actual measurement location.



Common problems

Too wide beam (collimator too large)

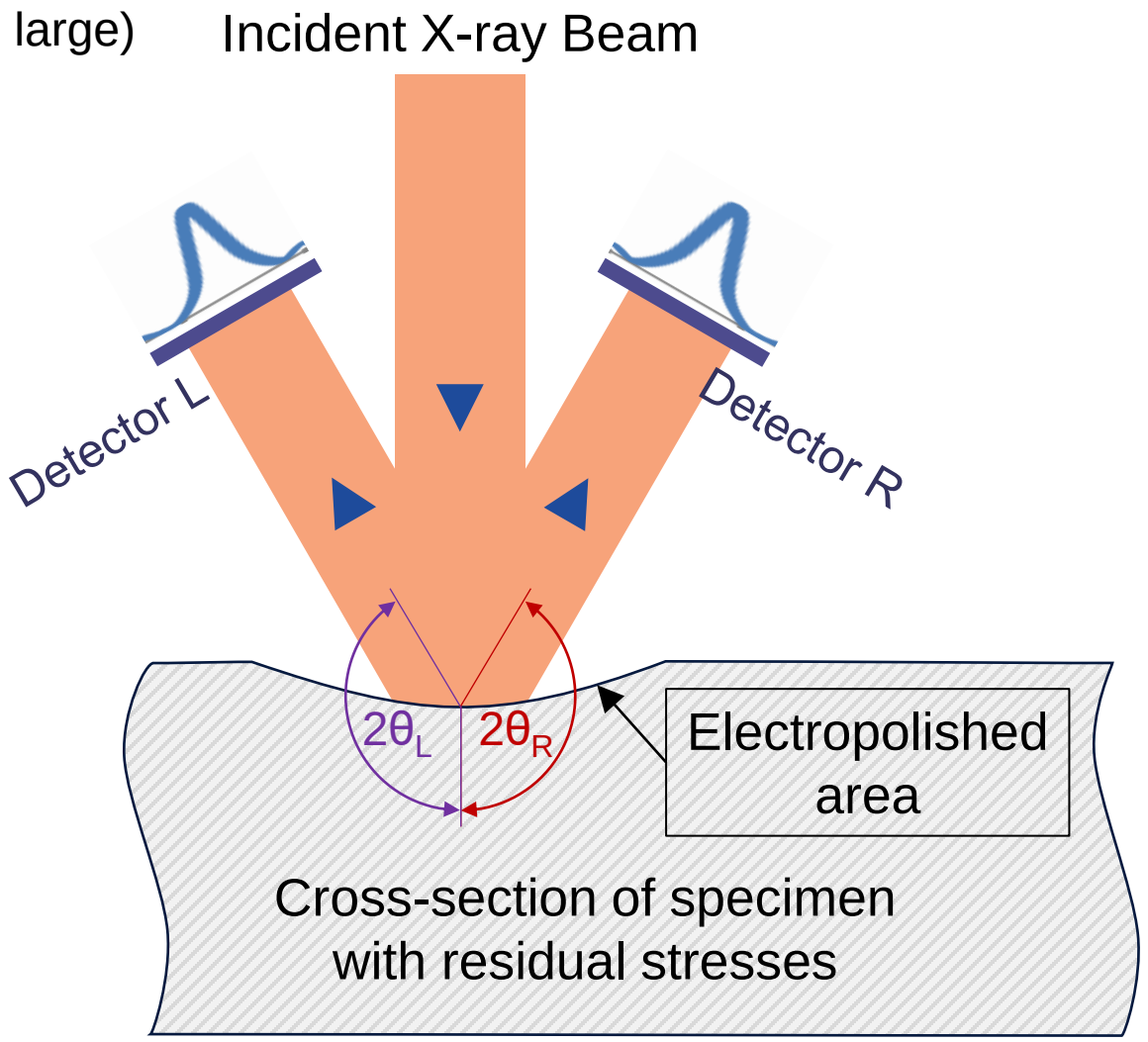
Problem:

Surface not flat
within gauge area.

Consequences:

Wide diffraction peaks,
large uncertainties

**Always choose a
suitable collimator size.**



Conclusions

- Implement correction for layer removal, but report both raw and corrected results
- Always electropolish at least 100 µm after grinding/machining
- Measure and report the actual measurement depth
- Always align the measurement point with the electropolished patch
  - Ensure perpendicularity
- Use appropriate beam size (collimator)

THANK YOU

Dr Jeferson Araujo de Oliveira

jeferson.oliveira@open.ac.uk