Gas loading for diamond anvil cells

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Types of failures:

- Crack propagation to gasket edge
  - Re may be flawed, examine Re for defects
  - Insufficient preindentation: higher pressure anneals cracks, go to higher P/thinner gasket
  - Too much medium or sample makes this more likely
- Hole shift blowout
  - Diamond alignment may be off
  - Hole drilled off-center
  - Instability due to too-thick gasket
- Hole expansion blowout
  - Too much medium: hole too big or thick, sample too big, or gas pressure too high
- Diamond failure
  - He (and Ne, but less often) propagates vertical cracks in diamonds
- Bridging
  - Gasket too thin to surround sample with medium, need thicker gasket or thinner sample

Recommendations:

Gasket

Material:

Herein I'm only considering Re gaskets, the best option for Mbar experiments. People seem to have strong opinions about their Re suppliers. Vitali Prakapenka only buys Re from Sigma-Aldrich these days, and our experience supports his opinion that their Re foil is stronger, finer grained, and deforms more evenly than Alfa Aesar's. At HPCAT they get their Re from another supplier that anneals it for higher strength. We've had crack-like failures with all three suppliers' Re, so this alone won't solve every problem.

Preindentation:

Stas Sinogeikin recommends preindenting to 1/2 of your target pressure. In terms of thickness, he gives 50-60 µm for target pressures of <20 GPa, 40-45 µm for up to 50 GPa, and 25-30 µm for 1 Mbar. The two rules don't necessarily correspond; if I'm going to a Mbar, and preindent to 50 GPa, I'm sure my gasket will be thinner than 20 µm. Also, these are rough general targets, not specifically for gas loading.

I have had best success in Ne loading of cells with beveled anvils with gaskets preindented to 20-25 microns thick. For 200-µm culets, I'll go up to ~30 microns thick. I have less personal

experience with larger culets. With more compressible gas/lower gas pressure, gasket should be thicker. However, thicker gaskets are more likely to deform in unpredictable ways.

Drilling:

Gasket holes need to be perfect: circular and well-centered. We have had success with both EDM and laser drilled holes as long as they're well-centered (though my experience is that the GSECARS laser drilling system doesn't make as round a hole at the ~20 μm diameter needed for 50 μm culets). Hole sizes need to account for shrinkage of the gas during loading. The goal is to have a hole ~1/3 the culet size after loading. Sergey Tkachev's rules of thumb for hole shrinkage are ~20% for Ne and ~50% for He when the gas loading system is operated at spec.

Diamonds

- Alignment needs to be near perfect: if tilted or shifted, the gasket hole will shift. Be sure to check alignment for slippage after preindentation and examine the sample from both cylinder and piston sides after loading.
- Diamond bending stabilizes gasket hole → if gasket hole has not approached within 10% gasket size of the culet edge by the pressure at which diamonds are significantly bent, gasket hole is most likely stable to the maximum pressure the diamonds allow
- If there's a lot of time between loading and experiment, sample must be checked for diamond damage. He and Ne can both propagate cracks in diamonds overnight. He can also escape over time.

Sample

The most likely problem for Mbar experiments is loading a sample that's too big. Gas loading works best when there's plenty of room for the gasket hole to contract inward. The sample is ideally ~1/2 the size of the hole after loading (perhaps 1/3 the hole size before loading for Ne).

If your sample can be made thinner, that gives you better chances of avoiding bridging while keeping the gasket thinner for higher stability.

Gas loading system variables

There are only a few things we can control at the COMPRES/GSECARS gas loading system:

- Target gas pressure in the chamber → at spec, 25000 PSI. Ne loading has been successful with as little as 17500 PSI. Gaskets loaded with lower pressure are more likely to be stable, but also more likely to bridge the sample.
- Rate of turning the screws to increase pressure → higher rate may prevent He damage, but slower rate may help the gasket deform more evenly if loading to higher P
- Target pressure in the cell → should be >12 GPa with He to solidify gas and reduce likelihood of diamond damage. Also, most blowouts will happen near the pressure of preindentation. If lower pressure data isn't important, Mbar cells should be compressed to >20 GPa to test stability.

Examples

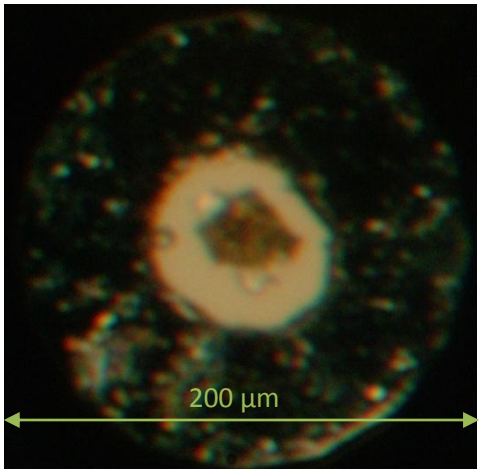

Figure 1: Successful loading of 200-micron culet cell with Ne at ~5 GPa. This sample was prepared for laser heating. Sample is on a NaCl tripod and there's a small ruby at culet edge.

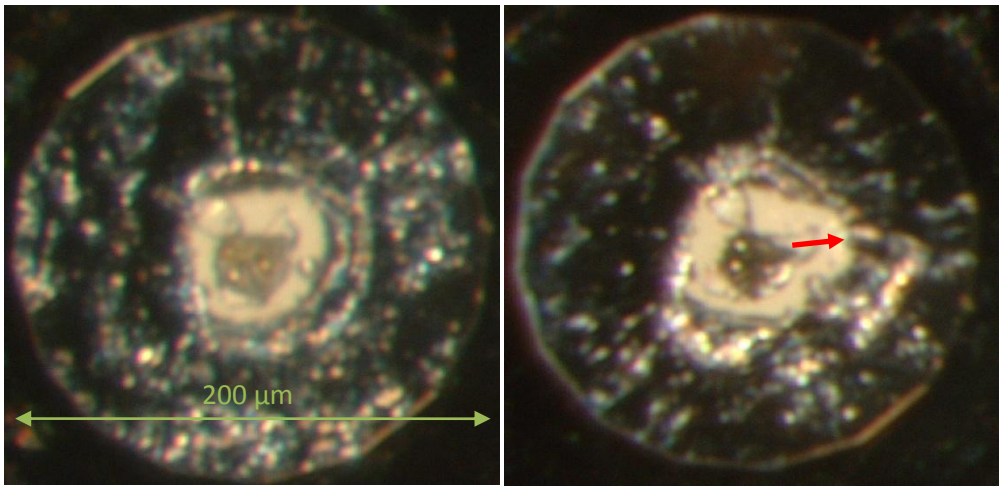

Figure 2: Almost unsuccessful loading of 200-micron culet cell with Ne. First image looks good after loading at 7 GPa. At 34 GPa, hole had expanded off to the right. Before this reached the edge, diamond bending was strong enough to hold Ne in.

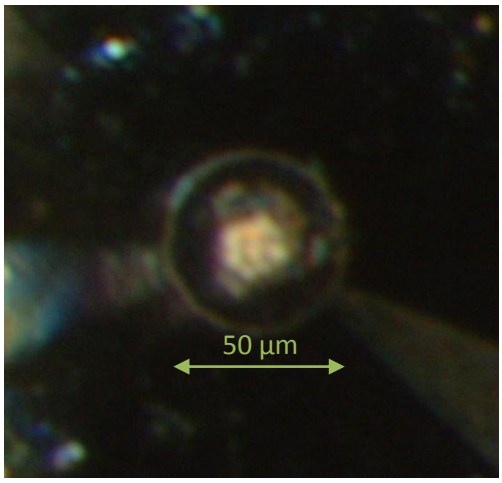

Figure 3: Successful loading of 50-micron culet cell with Ne at ~5 GPa. Original hole was ~25 microns and shrank to <20. This sample reached pressures above 2.5 Mbar.

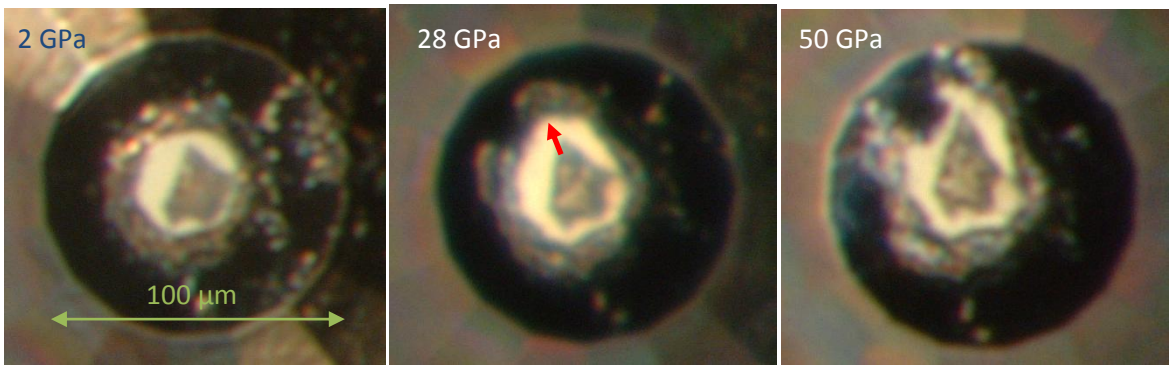

Figure 4: Unsuccessful loading of 100-micron culet cell with Ne. Immediately after loading, gasket hole looked good, but the hole expanded asymmetrically. This could be due to alignment, too large sample size, or other factors.

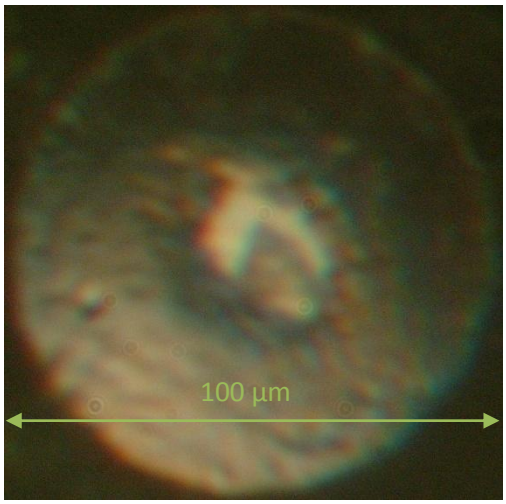

Figure 5: Successful loading of 100-micron culet cell with He, 20 GPa. This started with a similar hole to the above Ne-loaded cell, but the He contracted a lot more and the gasket was stable. In addition, the sample is smaller and the Re foil used for the gasket was finer grained.