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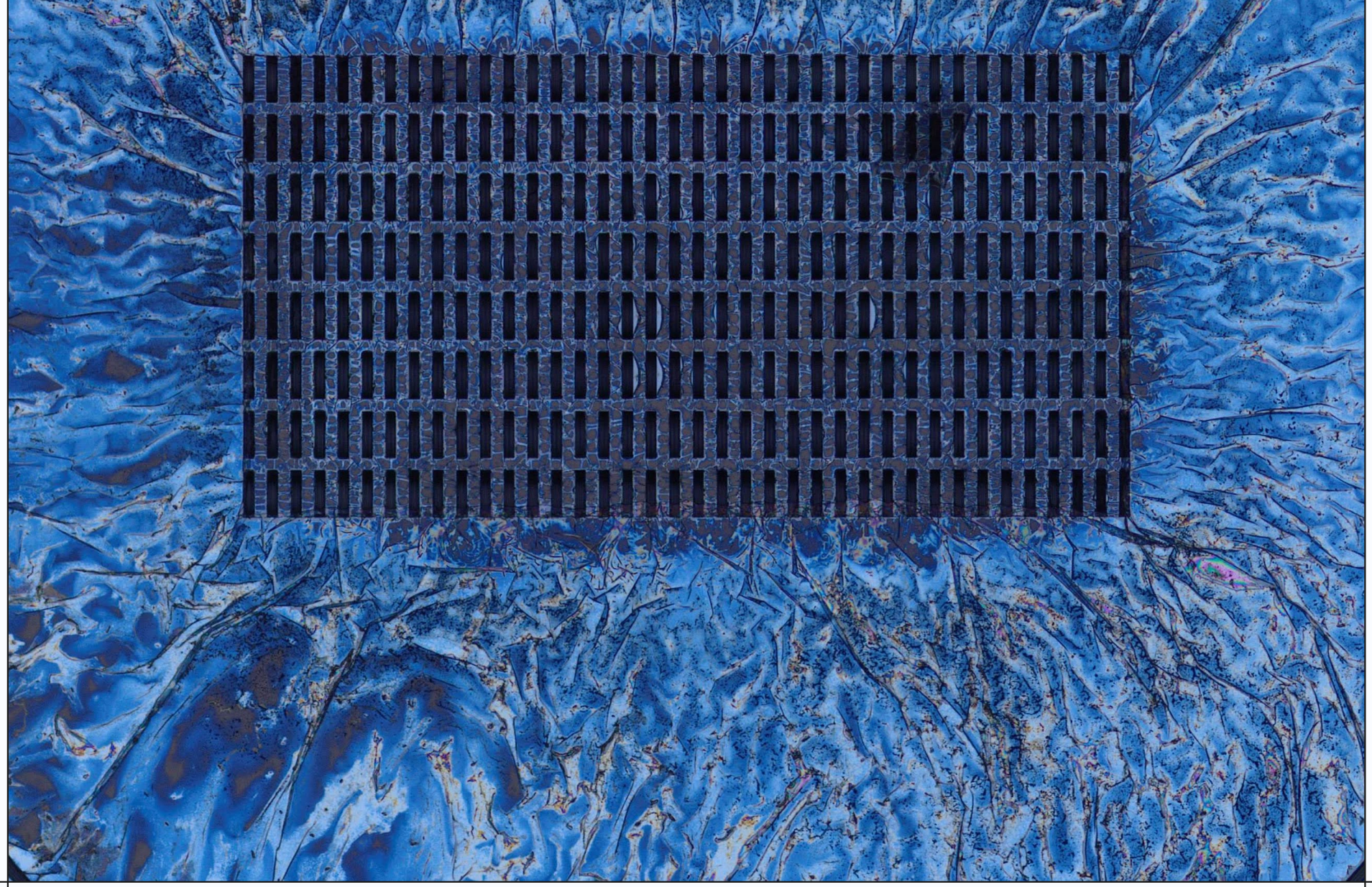

A Selection of Microscapes

Nemo Andrea
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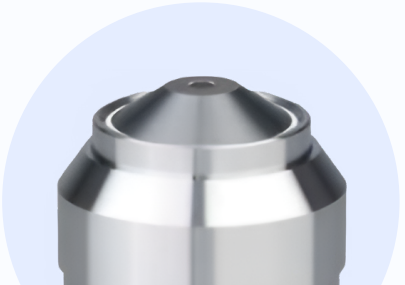

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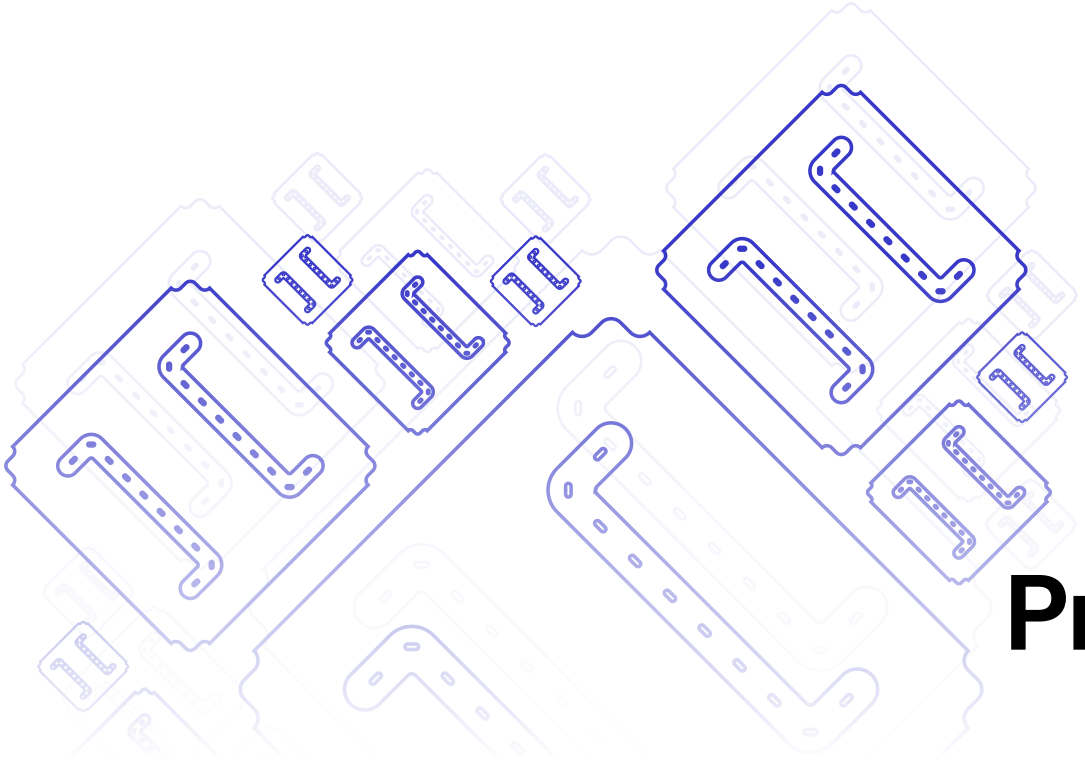


Preface

This photobook exists as a companion work to my doctoral dissertation. In this work, you will find a range of images all recorded with microscopes, that display a world and landscapes hitherto unseen. These microscapes are often encountered by chance, when an experimental process step has failed or otherwise produced undesired results. Perhaps we can take some sort of lesson in that; that things are rarely a complete failure.

The images presented here are all related to microfabrication: the world of computer chip fabrication and high tech cleanroom environments. The world of microfabrication is that of thin layers; a world that is closer to 2.5-dimensional than 3-dimensional. Extremely thin layers (down to 8nm) are deposited or etched onto a thin substrate, and the final devices are built up layer-by-layer. This device substrate, usually monocrystalline silicon that is appropriately referred to as a 'wafer', serves as an ultra-flat starting point for all subsequent processing steps.

The composition, pattern, refractive index, and position of each layer all contribute to the final observed colours in light microscopy images. The order and composition of these layers is indicated in each image in the sections marked with the symbol. The rightmost item in the list represents the topmost layer in the device. Most images have no intrinsic viewing orientation, and are best viewed as oriented on the page. For the small number of images that are best viewed in landscape orientation, you will find this indicated by the symbol. The microscope used to record the image is also listed for each image, indicated with the symbol. The images have only had brightness, crop and contrast adjustments, but are otherwise unedited. For each image, a scale bar indicates the size of the microscape being depicted — to remind the viewer that the landscapes present on these pages depict things that are small and smaller still.

On the topic of small scales: a micrometer (µm) is one thousanth of a millimeter, and a nanometer (nm) is one thousanth of a micrometer. The example scale bars below represent 1mm and 10 µm (1/100th of a mm) scaled up 100 times.

1mm (100x)

10µm (100x)

With that out of the way, I think you have all the tools you need to explore this book. I hope you will enjoy exploring these microscapes as much as I have.

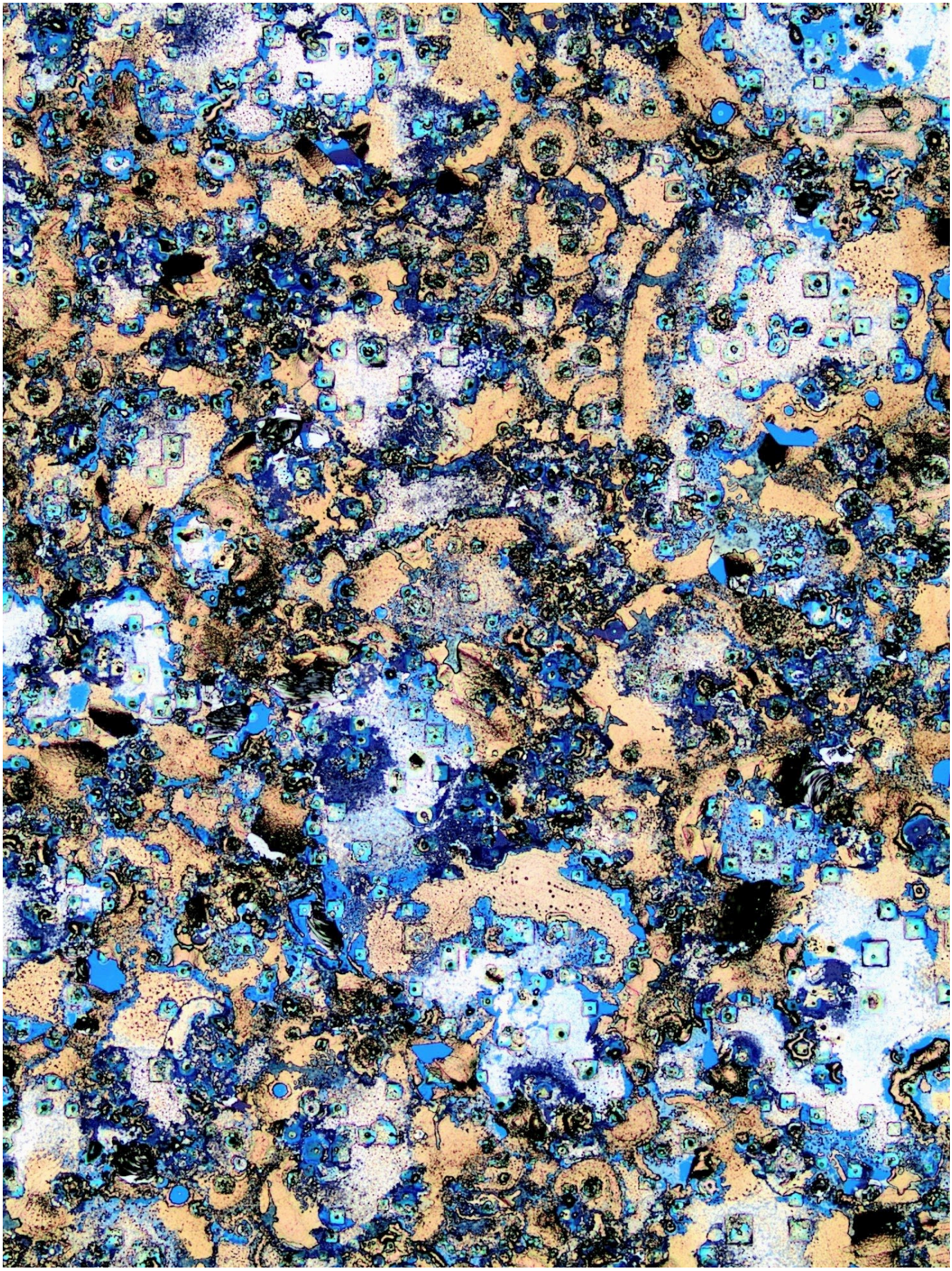
1. Inorganic

We start off our journey into the world of microscapes with images that contain only inorganic materials. Examples of inorganic materials are silicon, glass, silicon carbide, metals; essentially anything without carbon. These materials are the backbone of semiconductor technology, because the inorganic materials used are typically very stable, and can be tuned to have the extreme physical and electrical properties required for modern semiconductor applications.

Inorganic materials can have high internal stresses, and can be very brittle. If care is not taken to control the stress or ensure appropriate adhesion to prior layers in the microfabrication process, these materials can end up spontaneously releasing this stress by buckling (if the stress is compressive) or cracking (if the stress is tensile).

Three different imaging modalities were used to produce the images in this section and the next. Most images are recorded in brightfield reflection mode, which is where the light source illuminates through the same lens that is used to record the image. This is most similar to a conventional photograph with very flat lighting, just at a higher magnification. Darkfield images, on the other hand, illuminate the sample at a high angle, in which smooth and flat features result in dark regions in the image, and edges result in bright areas. This is similar to how dust particles in the air become visible when a bright streak of sunlight shines into the room from a window. Finally, there is differential interference contrast (DIC), which turns a difference of optical path length into observed contrast. While there is no easy analogy, in practice it also ends up highlighting edges and subtle surface imperfections.

While you will find that the images are full of colour, this colour is often not caused by absorption, but by structure. That is, the colour is a result of how light interacts with the geometry of the layers on the substrate. This is because many of the non-metallic layers (e.g. silicon nitride, aluminium oxide) are actually transparent, and the observed colour is due to thin-film interference. Changing the thickness of the layer would change the observed colour in a very predicable fashion. Changing the angle of observation relative to the surface will also change the observed colour, which is readily observed when handling and tilting the sample.



The Great Pyramids of Nickel

100µm

Nickel is a very good diffuser in silicon. In an attempt to find ways to grow graphene on a silicon substrate, a thin film of nickel was deposited on a SiN-coated silicon wafer. The silicon nitride was unable to prevent diffusion of nickel into the silicon when heated to 900°C in a CVD chamber for graphene growth. The resulting alloy is likely eutectic; evaporating at a lower temperature than the individual components. The evaporation followed the crystal orientation of the silicon substrate, forming pyramidal pits visible as squares.

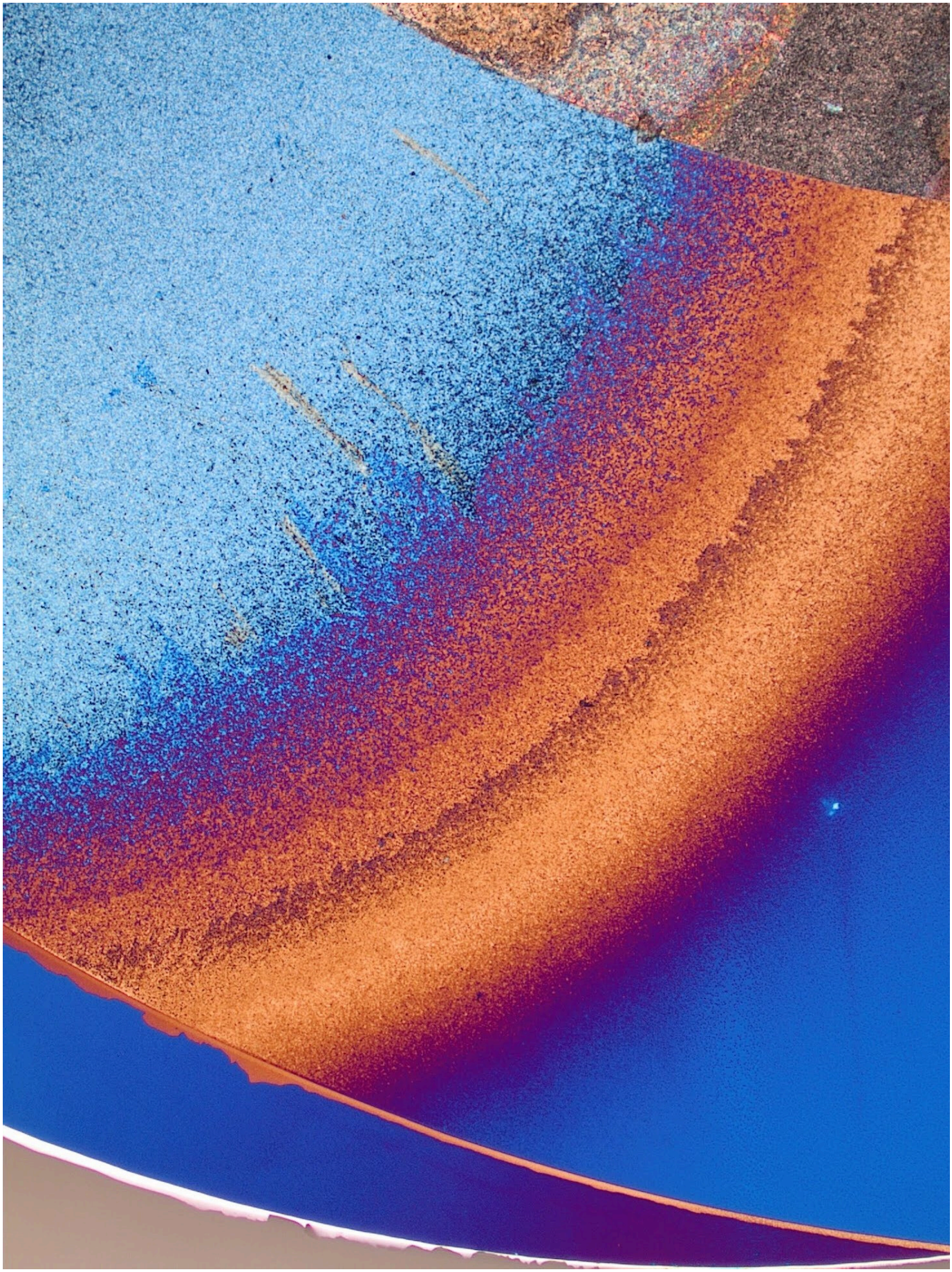
Olympus BX51



The Root Network

100µm

Another example of the high diffusivity of nickel in silicon. Here a thin film of nickel and molybdenum was deposited on a silicon nitride-coated silicon wafer, and patterned in circles. After a 900°C CVD process, a brown alloy of all aforementioned materials is formed, where in areas without the metal a dendritic structure formed underneath the silicon nitride. This is likely the nickel (and molybdenum) that is able to easily diffuse deep into the silicon lattice in all directions.

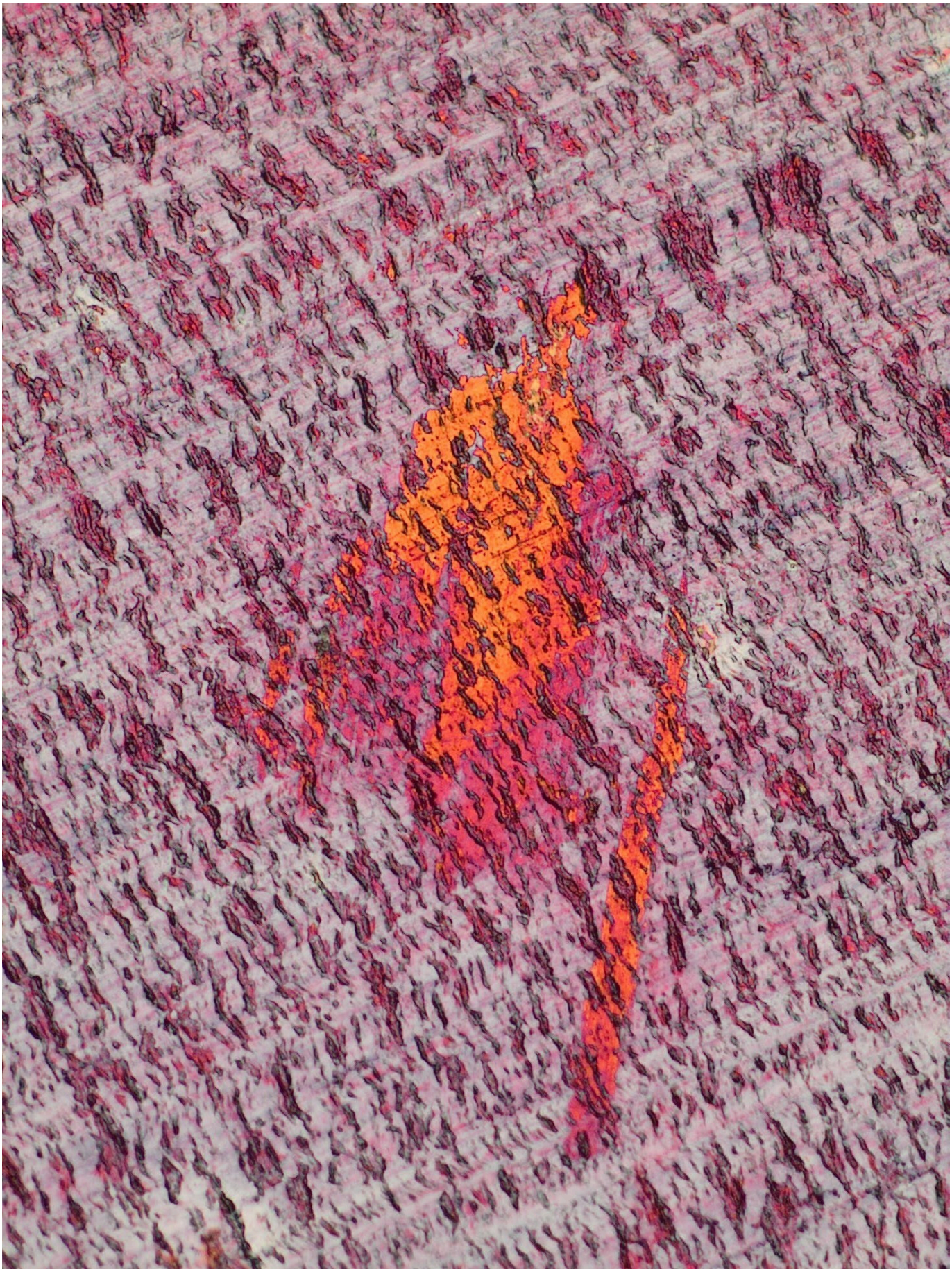


The Molybdenum Rainbow

90µm

The primary colourful gradient in this image is a result of a failure to remove all of the molybdenum from the wafer surface. The molybdenum was used in a CVD process at 900°C to grow graphene, and had to be removed after the graphene had formed. The hydrogen peroxide left half-etched molybdenum/molybdenum-oxide behind. In other areas of the wafer (not shown), all of the molybdenum was easily removed, indicating that this particular area may have had particularly high quality graphene preventing etchant access.

Olympus BX51

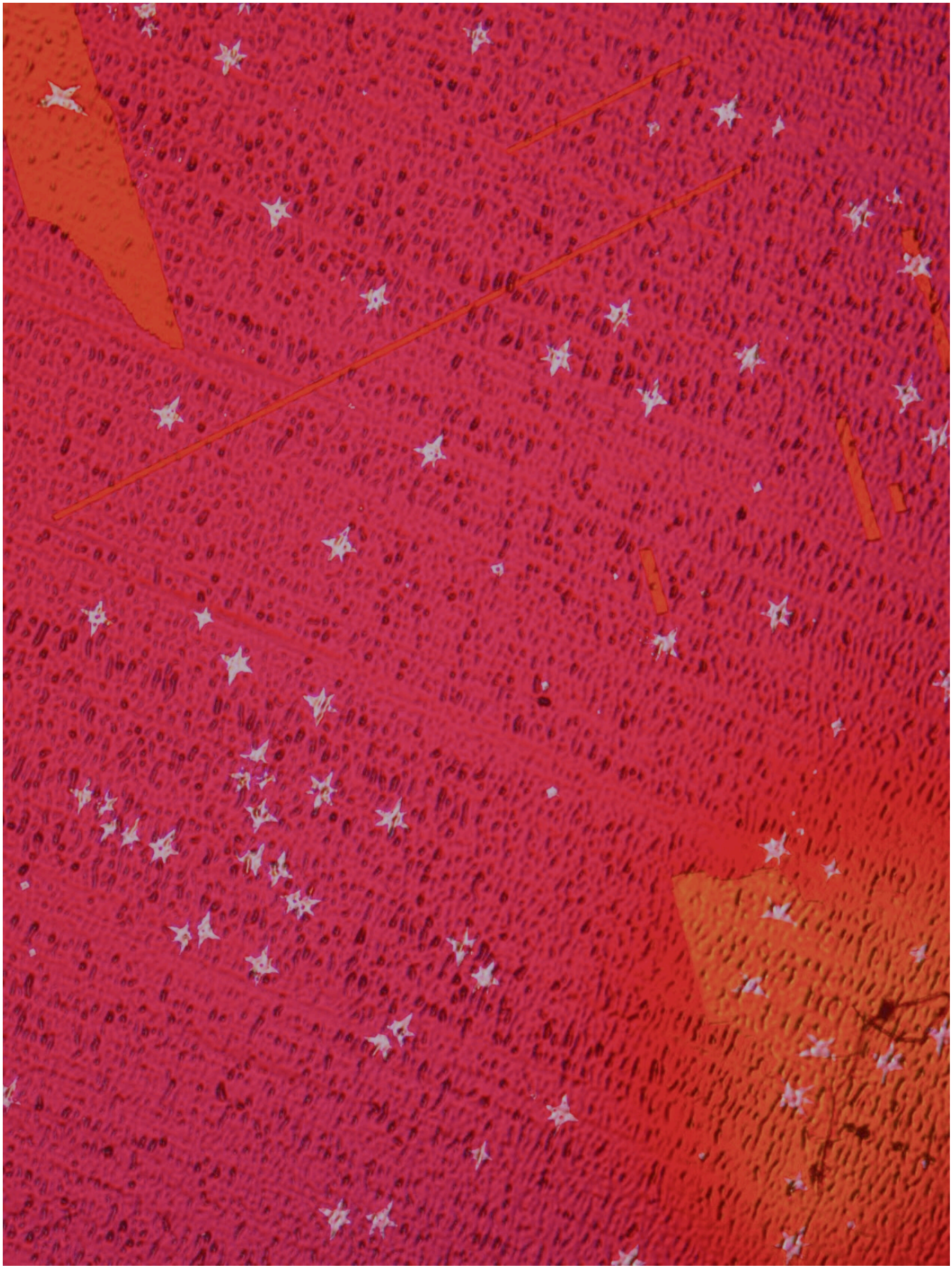


Lonely Oxide Island

50µm

A piece of copper foil after a graphene CVD process. To test for the presence of graphene, the substrate is heated in air. If graphene is present, the surface will remain pristine, and if graphene is absent, an oxide layer will form which can easily be distinguished by a colour change. Most of the texture in the foil likely stems from the foil's manufacturing process.

Leica DM 2700

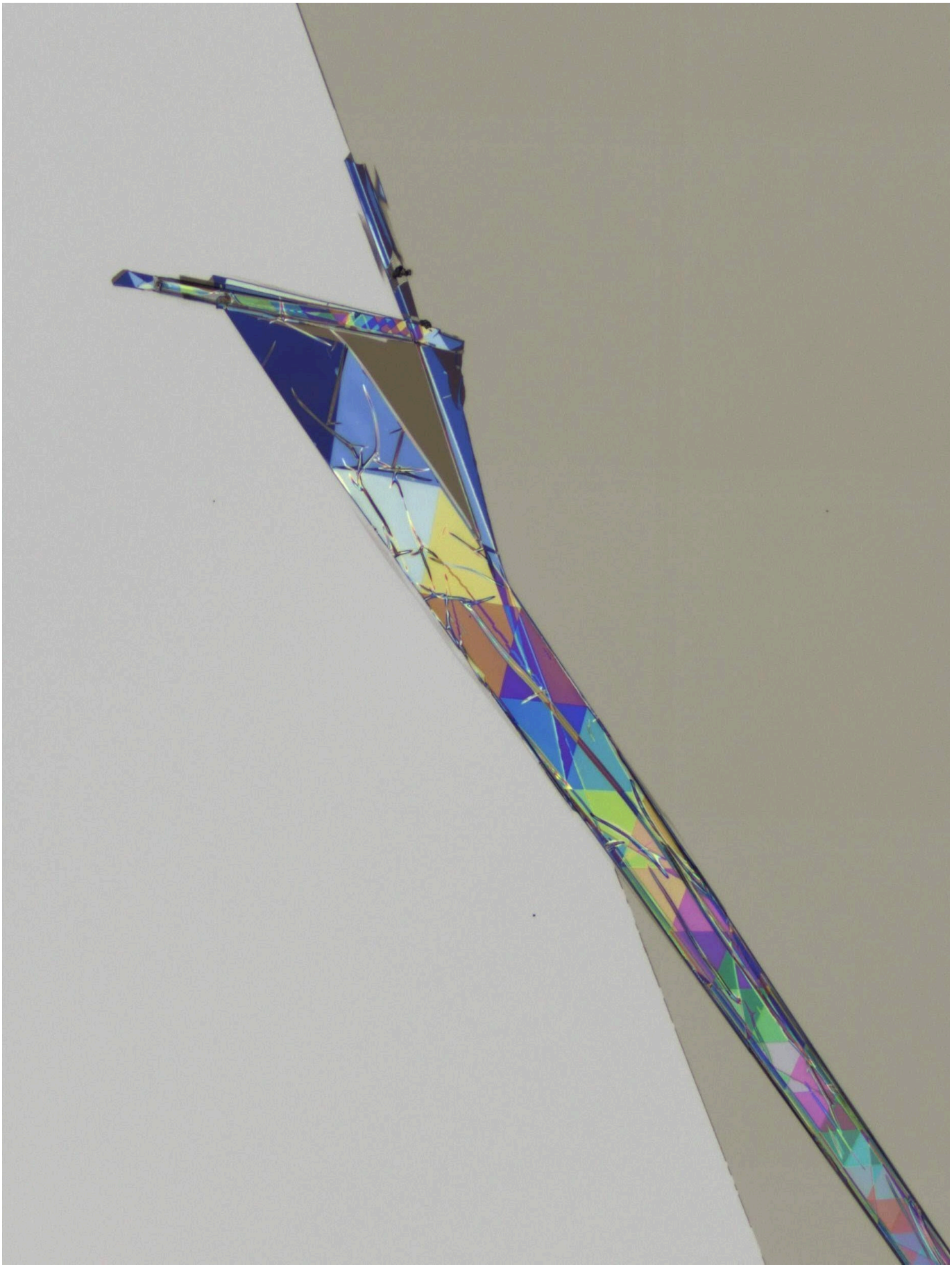


Starry Night

50µm

In attempts to grow graphene with large crystal size, we set out to decrease the graphene nucleation density on the copper surface. To check this, we would heat up the copper foil (after graphene CVD) in air, where areas not coated in graphene will change colour due to oxidation. Here we see many small star-shaped graphene crystals that were—much to our dissapointment—not very hexagonal. The star shapes indicate that this growth process was likely diffusion-limited.

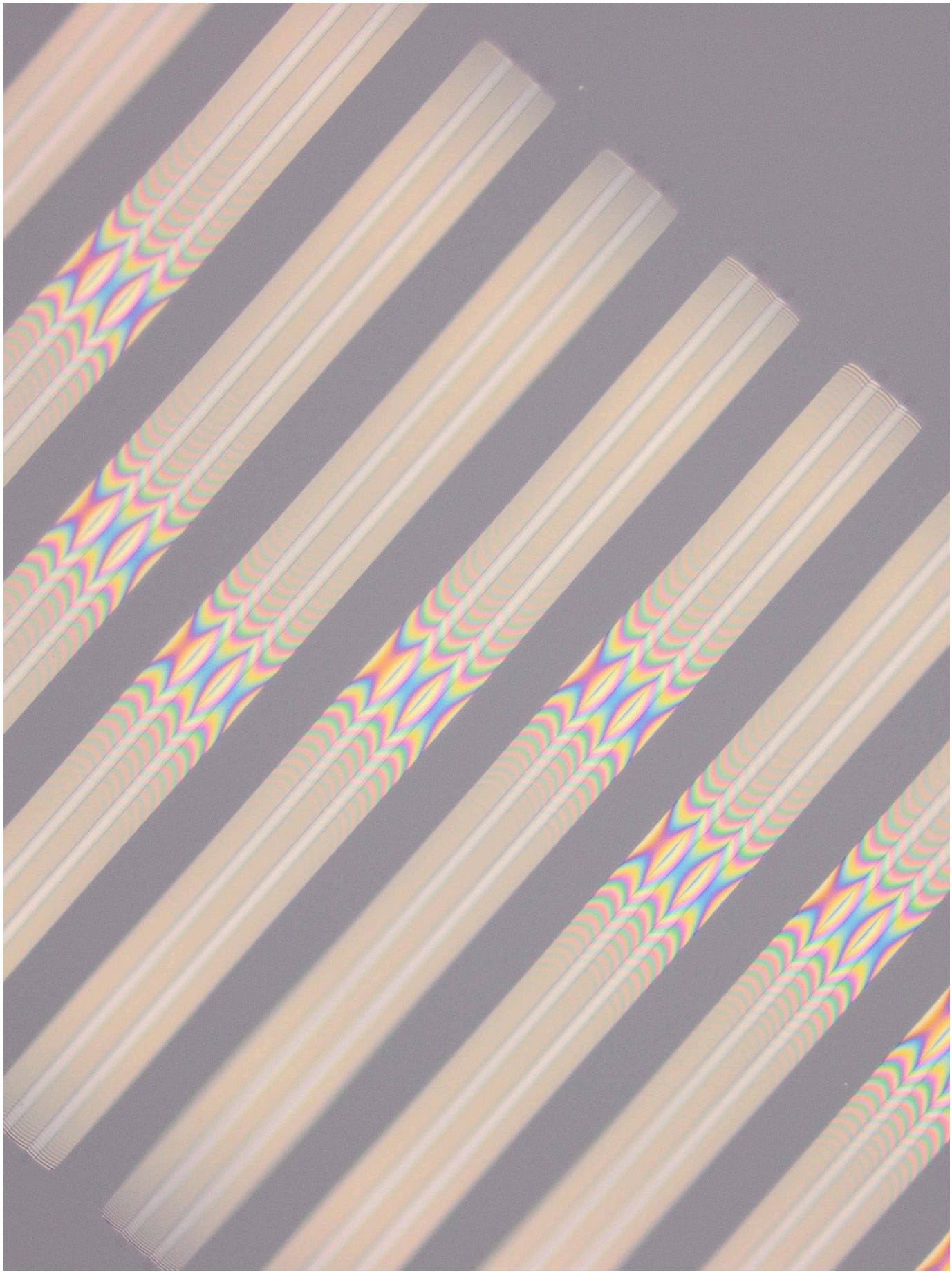
Leica DM 2700



The Banner of House Thin Film

100µm

To remove an inorganic layer one can deposit it on top of a patterned polymer layer, and then remove this organic layer in solvent, to ‘lift-off’ the deposited material. If all goes well, this material does not redeposit onto your substrate. Clearly, luck had taken a day off in this image. The redeposited aluminium oxide thin film has folded onto itself many times — with each number of folds producing a separate colour, according to the principle of thin-film interference.

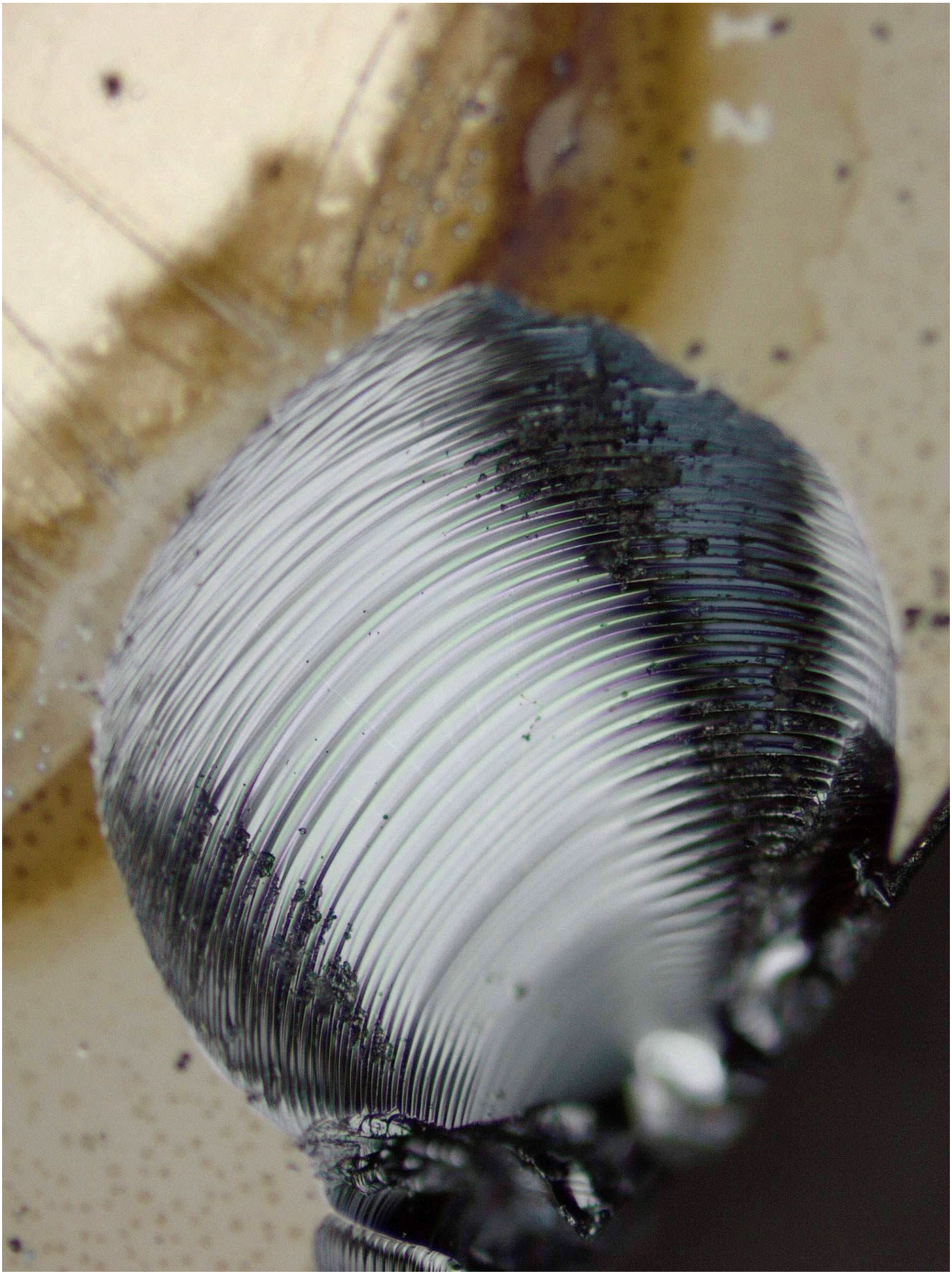


The Edge of Adhesion

100µm

These devices were made on top of standard coverglass pieces, and we are looking *through* the substrate. A series of nanochannels are visible that are starting to delaminate from the coverglass surface, producing a colourful interference pattern due to the varying distance between the reflective molybdenum and the coverglass surface. The adhesion between various ALD layers and the substrate was reduced by hydrofluoric acid vapor treatment, which proved to be challenging to control reproducibly.

Olympus BX51

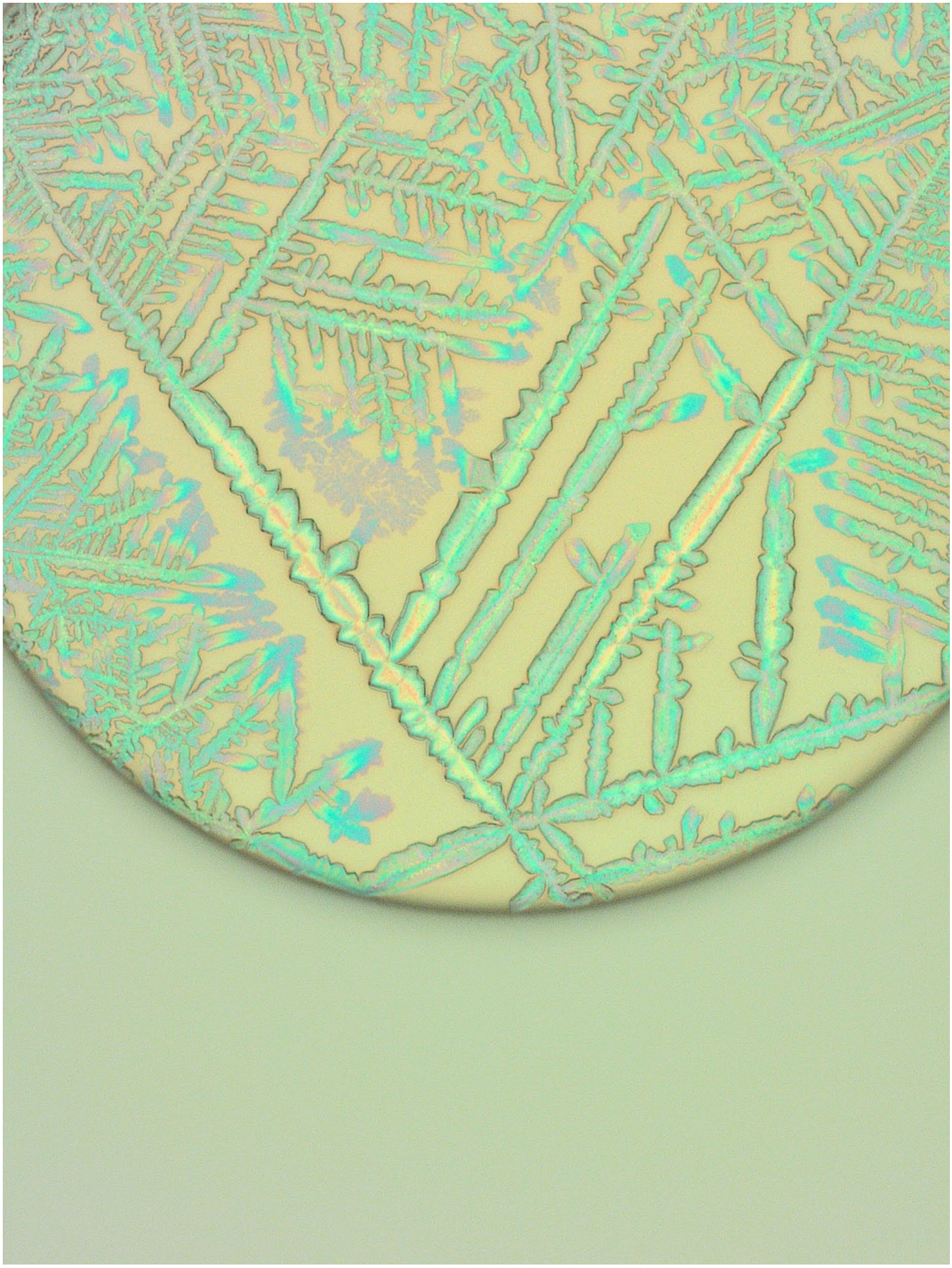


Chip Off the Old Block

50µm

The most common base substrate for semiconductor fabrication is monocrystalline silicon. The 0.5mm thick wafer measuring 10cm in diameter can be considered a single crystal. This single-crystalline nature means that the substrate will fracture, rather than deform plastically when mechanically pushed beyond its limits. When snapping tabs of silicon off the substrate, or when handling pieces of silicon with hard tweezers, it is not difficult to chip off a piece from the crystal. Good for microscale arrow-making.

Olympus BX51

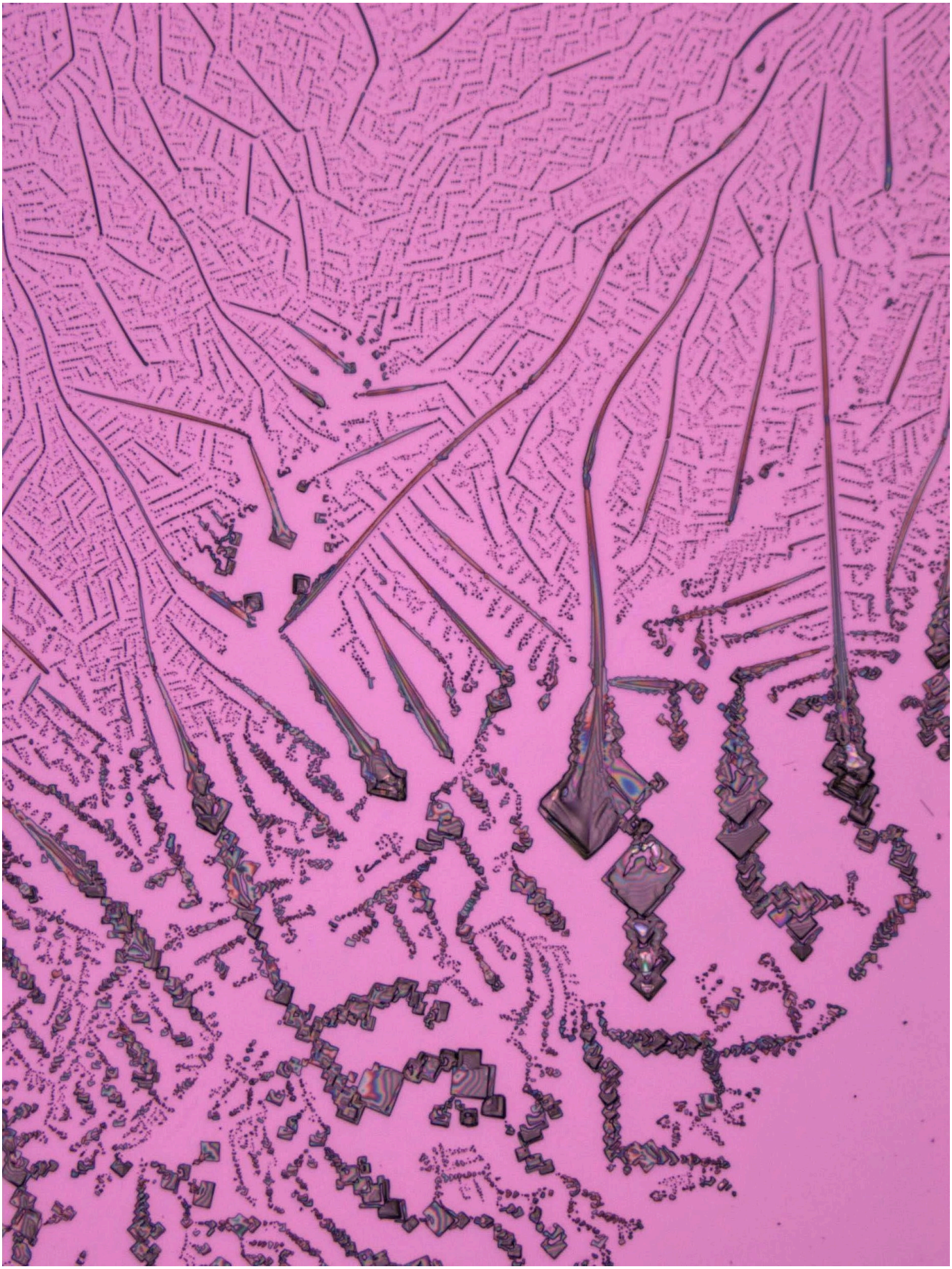


A Little Early for Christmas

50µm

While ‘glass’ is in its most basic conception a combination of silicon and oxygen, the ratio, density and impurities present in a glass mixture can radically change the properties of a glass layer. Here, a SiO_2 layer was deposited with a high amount of hydrogen present in the film. When etching this layer in a buffered hydrofluoric acid, the etch process occurs at such a rapid pace that ammonium salts can form and precipitate out of solution. These salts can then form intricate crystals on the substrate.

Olympus BX51

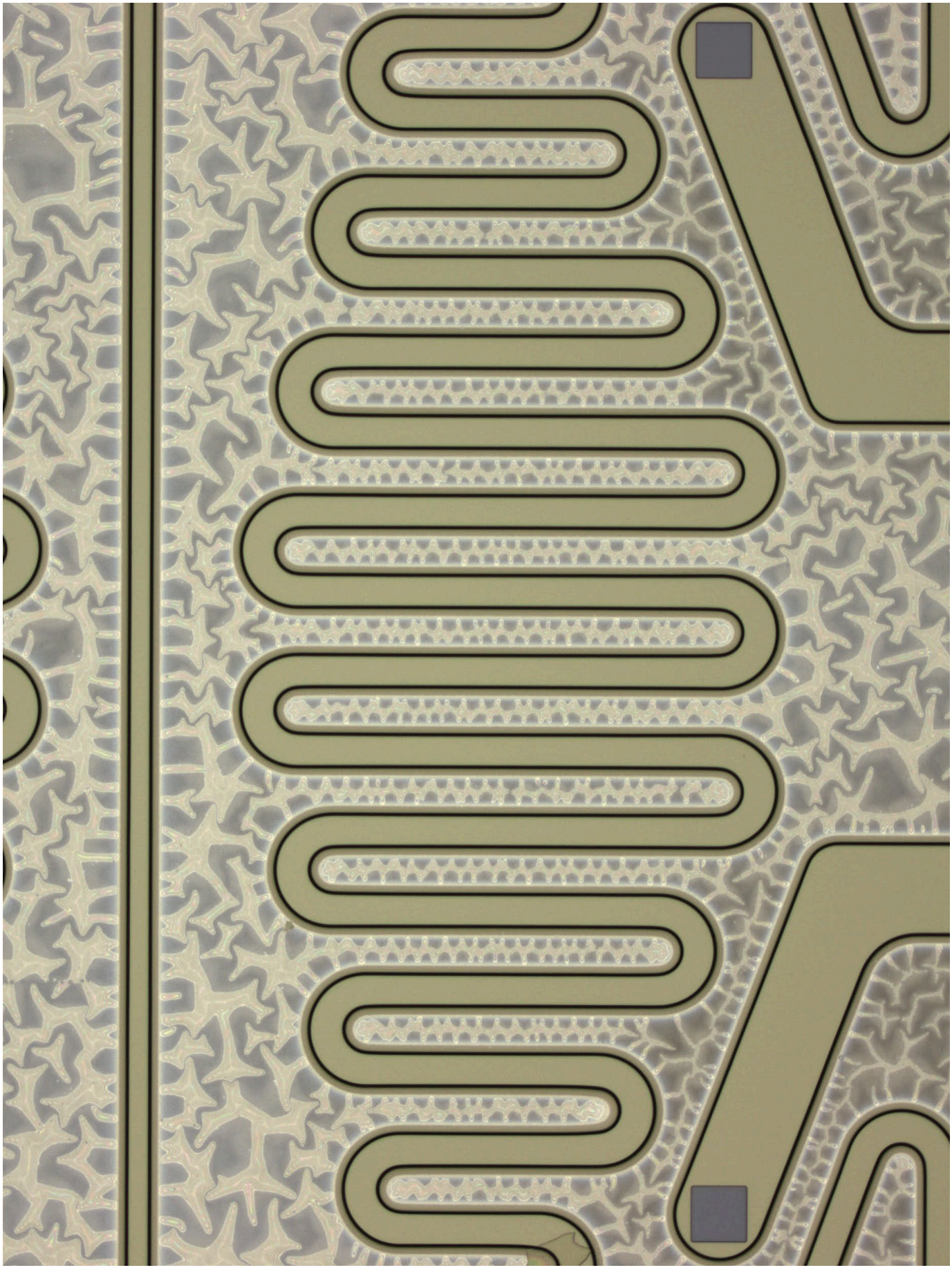


Salty Tadpoles

100µm

Silicon can be rapidly etched in highly alkaline solutions such as concentrated potassium hydroxide. This is a highly cost-effective method to etch large parts of a silicon wafer, and will only etch silicon along specific crystallographic planes. This can be used to form very specific pyramidal holes in the silicon. The downside of etching in a solution with a large amount of ions is that you need to ensure the substrate is rinsed properly afterwards — else you may find salt precipitating onto your freshly etched surface.

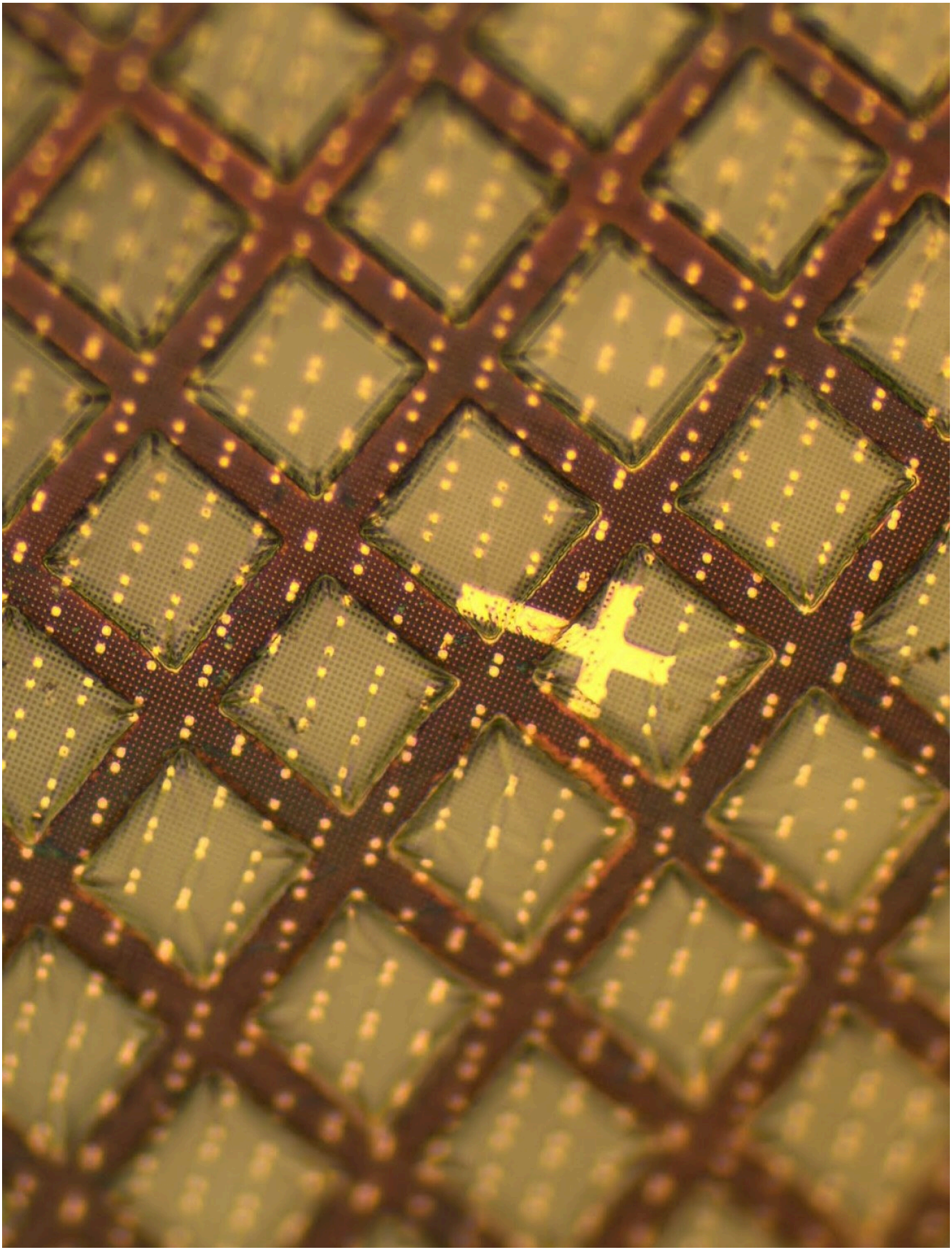
Olympus BX51



A Stack of Many Surprises

100µm

Some fabrication failures are easily interpreted, while others—like this one—are harder to diagnose. A SiO_2 layer was etched in hydrofluoric acid on top of a molybdenum layer. After trying to remove the molybdenum with a plasma-based process, these blister-like structures ended up forming. Whether it is the molybdenum film delaminating, residual SiO_2 masking the molybdenum, or whether the layer below has been etched by the hydrofluoric acid, one thing is clear: the layers in this stack are trying hard to be somewhere else.

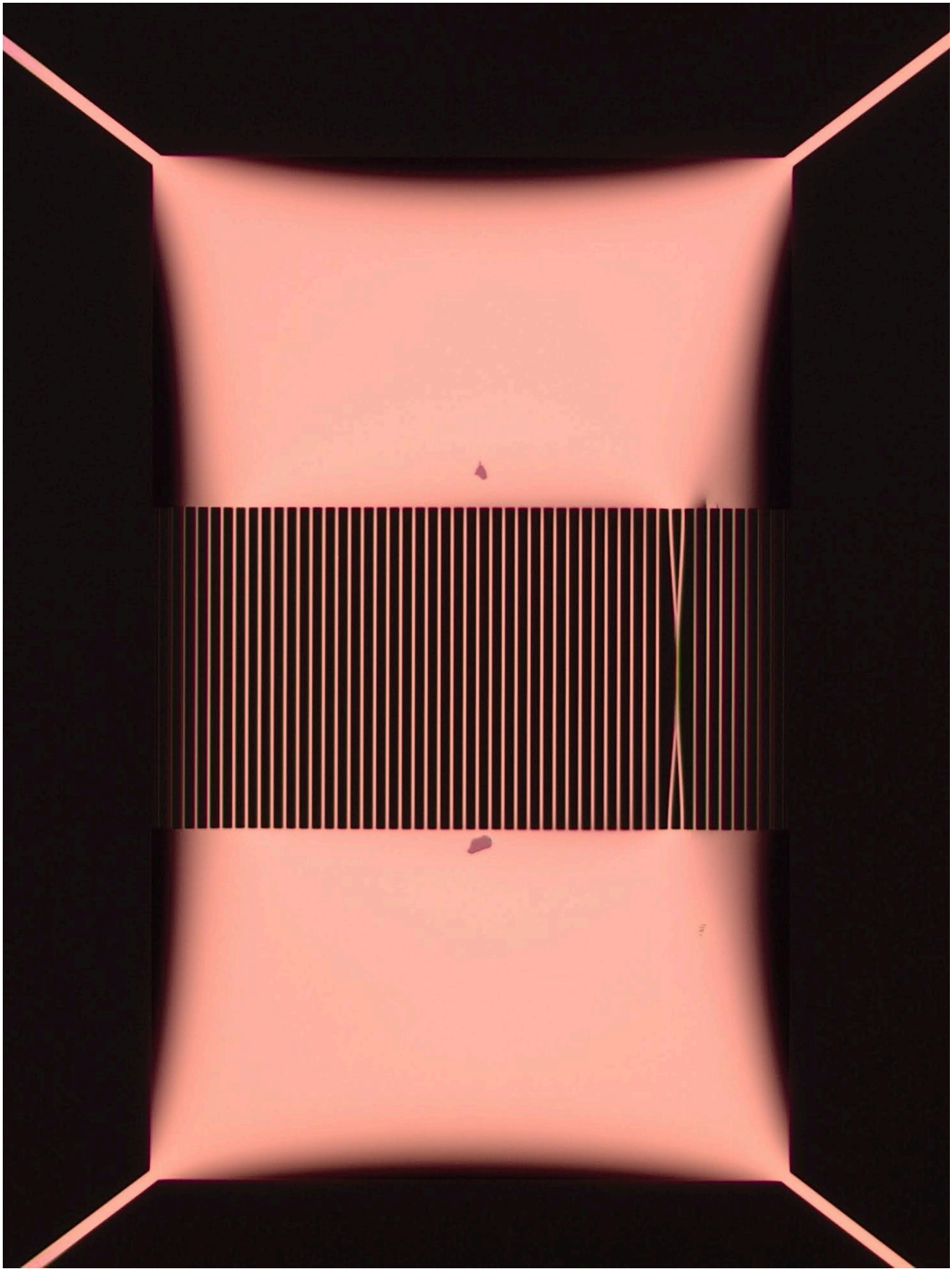


Aurea Mediocritas

100µm

In an attempt to find a middle ground between over-engineered and fully stochastic formation of small liquid pockets, we wanted to form thin gold structures on graphene-coated electron microscopy grids. These grids are the standard substrate for transmission electron microscopy. We used a silicon nitride shadow mask and evaporated gold through it, forming small (around 2 micrometer) gold structures on the grid. Unfortunately, our attempt at finding an aurea mediocritas was not very succesful. Perhaps *'fool's gold'* would have been a better title for this one.

Leica DM 2700

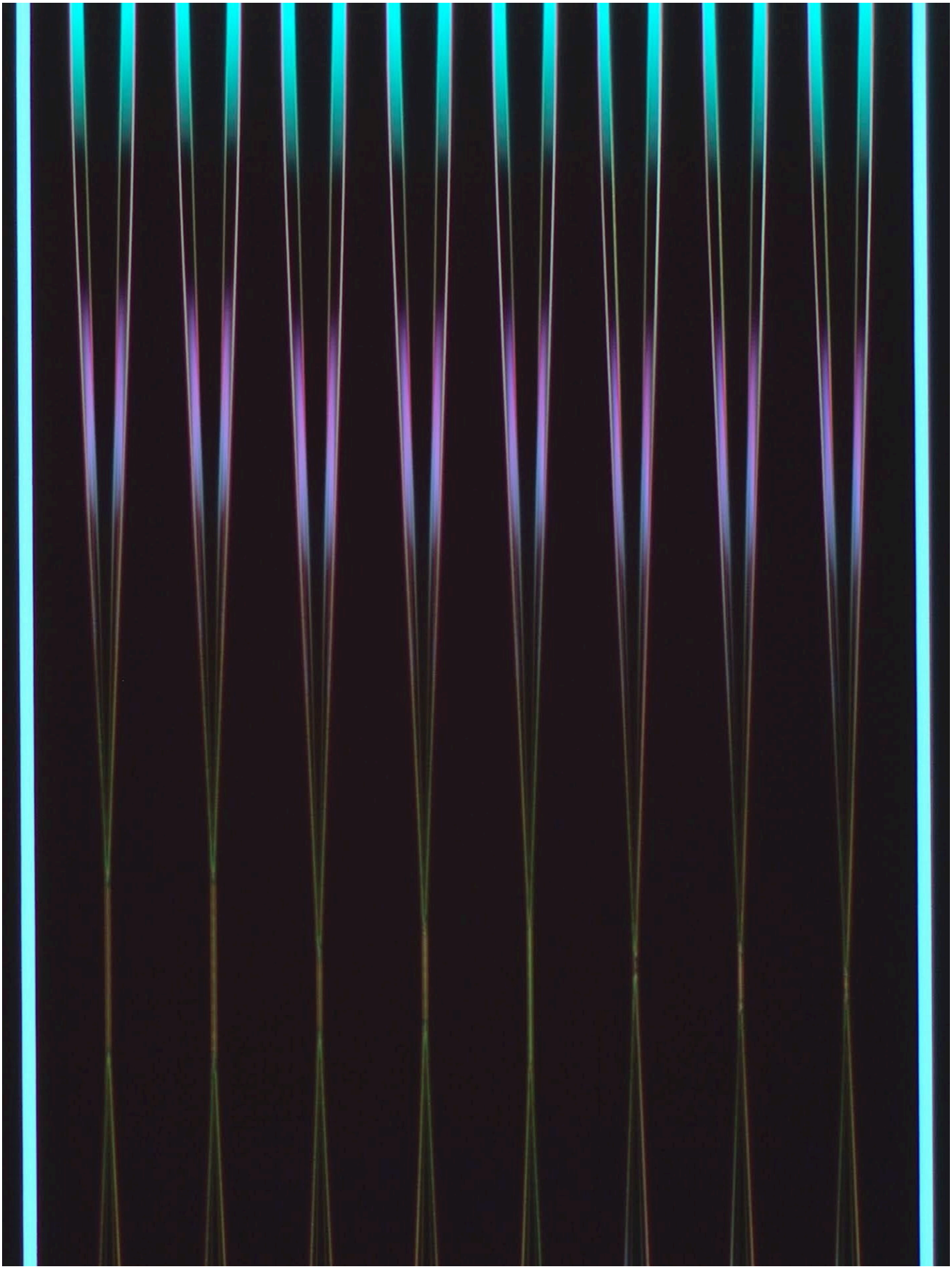


Keeping Me in Suspense

100µm

For another gold evaporation project we needed to make some suspended stencil masks. Here, a 300nm thick silicon nitride membrane is suspended over an open cavity, held taut only by four narrow tethers. It is truly remarkable how such a thin material can be so strong. The resulting substrate can be handled with some care without these ultrathin membranes rupturing. Here we evaporated gold onto these membranes, which resulted in warping of the membrane due to the intrinsic stress of the evaporated gold layer.

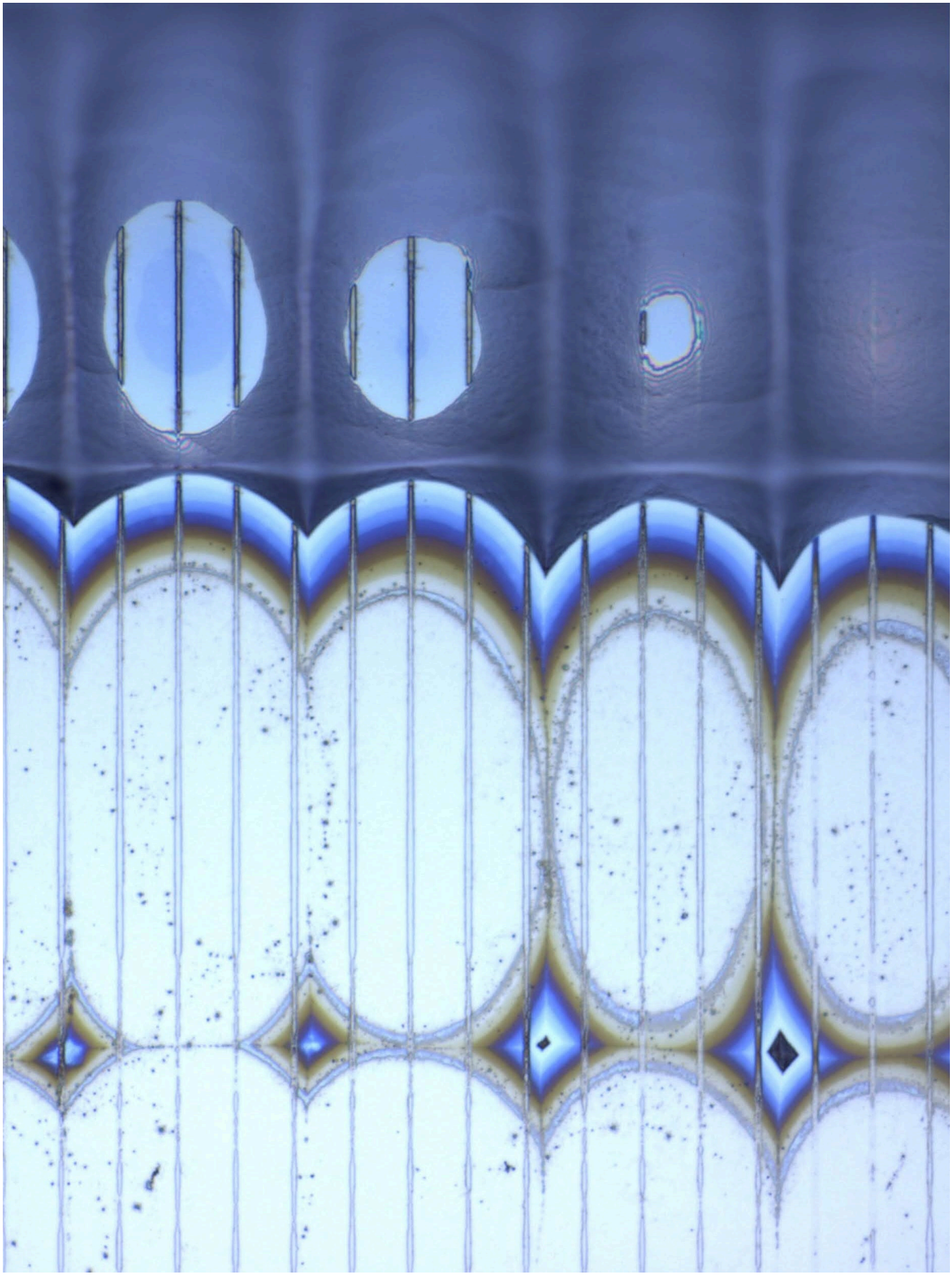
Olympus BX51



Intertwined

40µm

For the same project as Inorganic 13, we needed to obtain narrow parallel strips of silicon nitride. Here we see these narrow strips without a gold coating. Unfortunately, the thin silicon nitride strips ended up finding a buddy and sticking together. Once entangled, they would no longer come unstuck. I'm sure there is someone out there making clever use of this kind of effect.

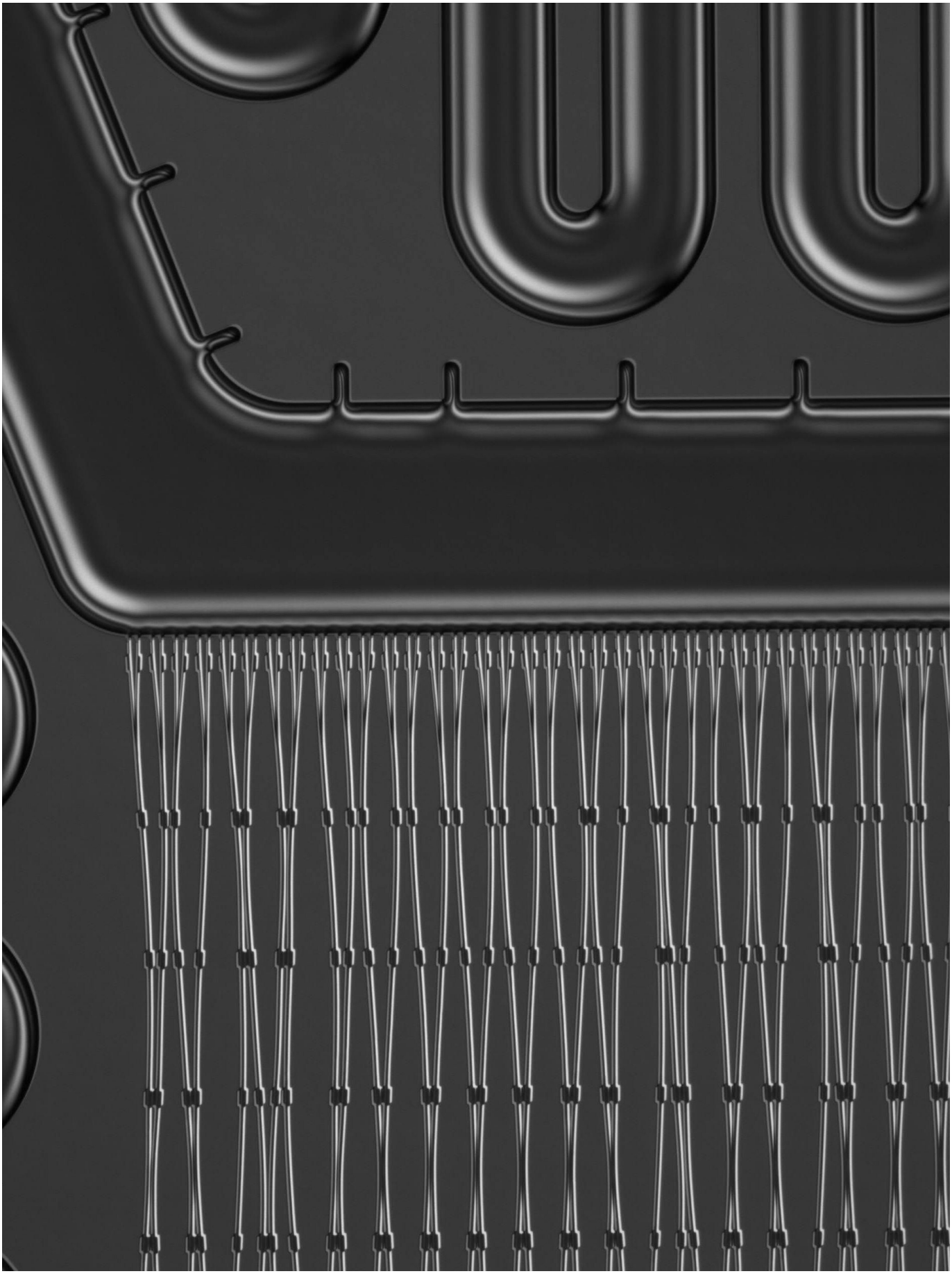


Layercake

40µm

Here we are looking at a silicon substrate from an unusual angle: through the wafer from below. The silicon was etched with deep reactive ion etching (DRIE), and the stop layer—whose task it is to etch only very slowly or not at all—was not doing a very good job. The silicon nitride layer ended up getting etched into a gradient of thicknesses, forming the blue-to-brown gradient. The etch process eventually terminated on the silvery nickel layer, which is completely inert to the DRIE etching radicals.

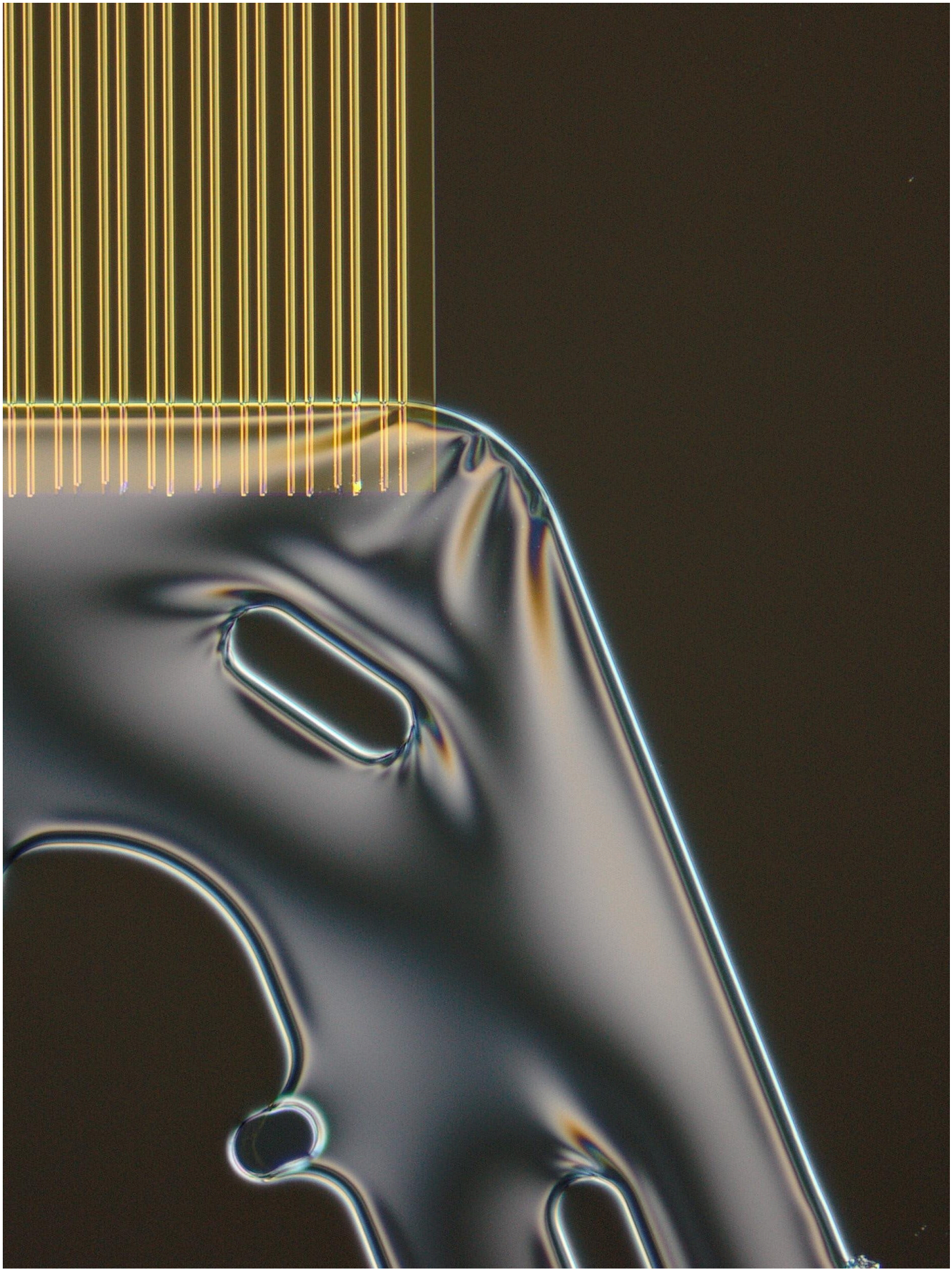
Olympus BX51



Intertwined II

100µm

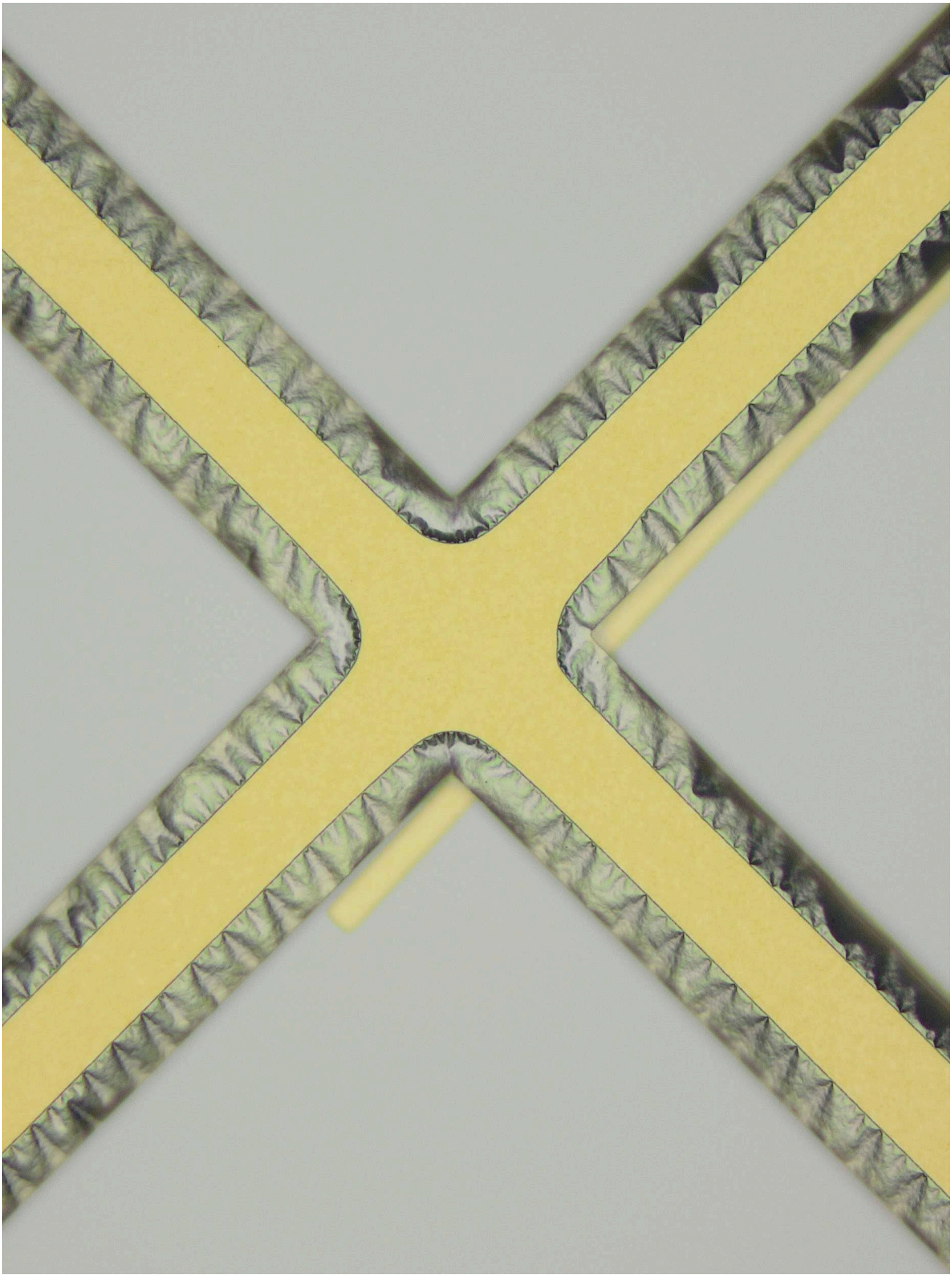
While not suspended, here too we faced the challenge of keeping long thin features on the device from getting too intimate with a neighbour. The origin of the problem here lied in the fact that the substrate below the channels (aluminium oxide) would be etched by the chemicals used to develop photoresist. The erratically tangled lines bring some much-needed liveliness to the otherwise very sterile differential interference contrast image.



Stress Lines

100µm

When imaging with differential interference contrast, small differences in surface topology as a result of stress become apparent. Here we see some free-standing silicon dioxide microchannels that were deposited on top of a highly contractile polymer layer. After the polymer was removed, the glass layer stays in that stressed geometry — preserving it for eternity. I suppose that there might be a lesson about taking more time off in that.



Micro Awnings

100µm

Some etch processes are *isotropic*, i.e. they will etch in all directions at approximately identical rate. When a material is etched in an isotropic fashion, the masking layer will often get undercut—as material is also underneath the mask as the etch progresses. Here, an oxidised molybdenum film was on top of a glass layer that was isotropically etched. The resulting molybdenum film ended up wrinkling in the areas where there was no more glass to support it. A good place for a bacterium to hide from the sun.

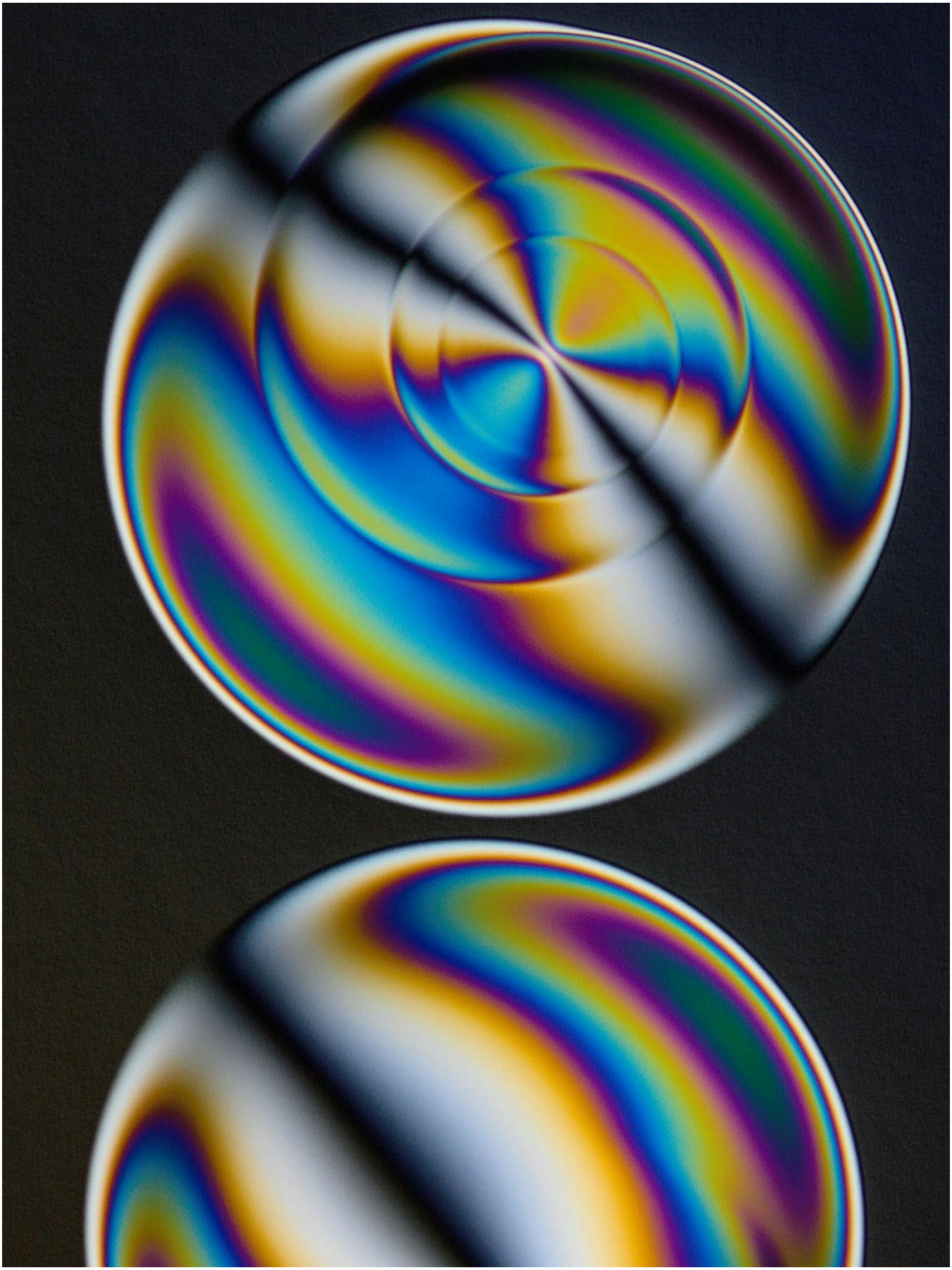
Olympus BX51



Exoskeleton

100µm

Most thin films have non-negligible stress after depostion. This stress can be either compressive or tensile. This stress can sometimes be desirable, while in other instances low stress might be preferred. When making free-standing fluidic channels, lower stress is probably better. In the image above, the glass layer did not meet low stress requirements, and ended up cracking in many places as its supporting polymer layer was removed.

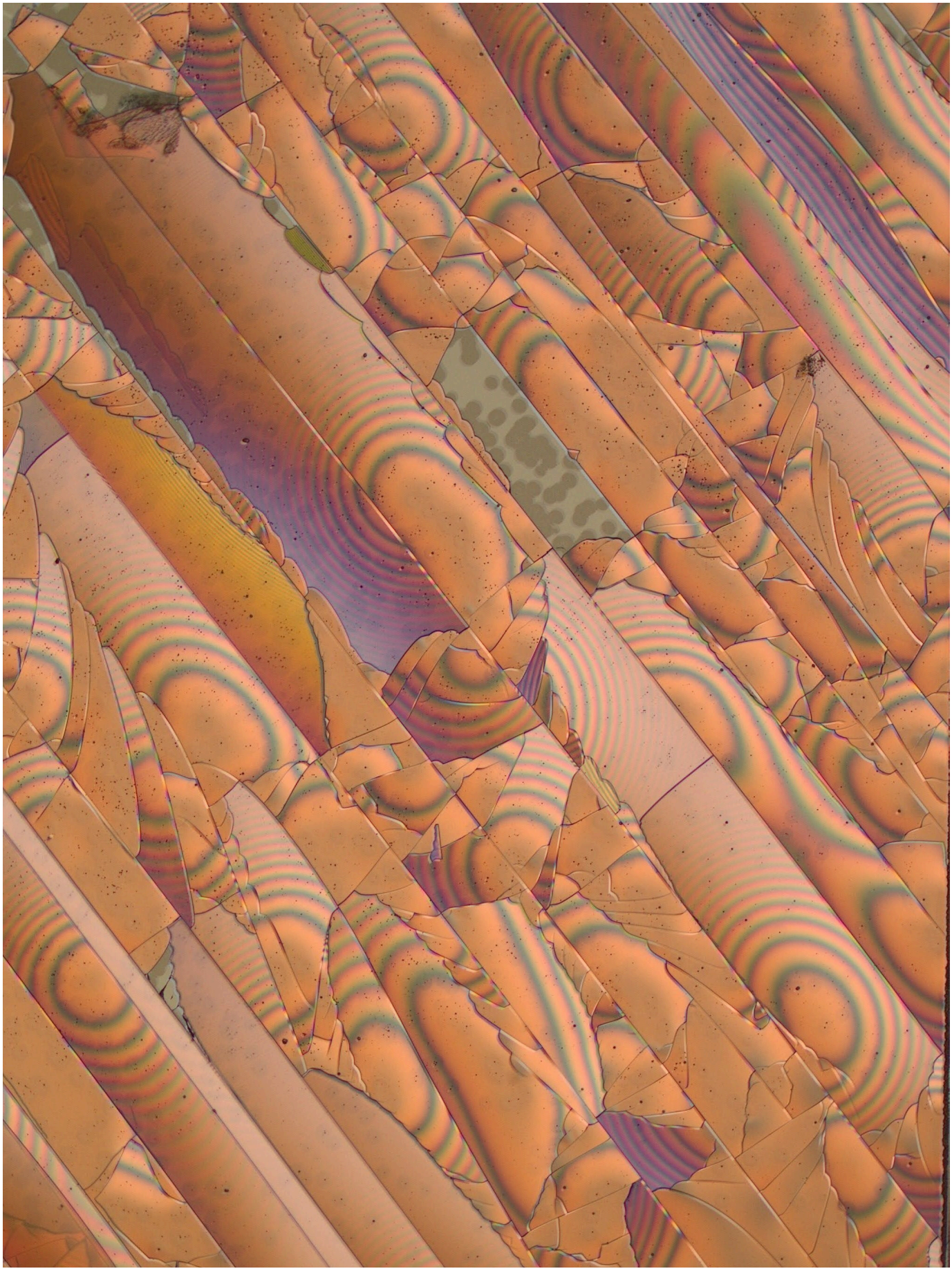


Bad Skincare Routine

300µm

While we are on the theme of layer stress, the image above shows what happens when a layer with very high stress is deposited onto a substrate with a poor adhesion interface layer. In some places, the amorphous silicon layer ended up spontaneously delaminating into (spiral) blisters. It had another trick up its sleeve: when I heated up the layer in an extended oxygen plasma treatment it turned itself into a layer of fine dust. I'm personally more fond of tricks that don't result in me having to clean the machine afterwards.

Olympus BX51

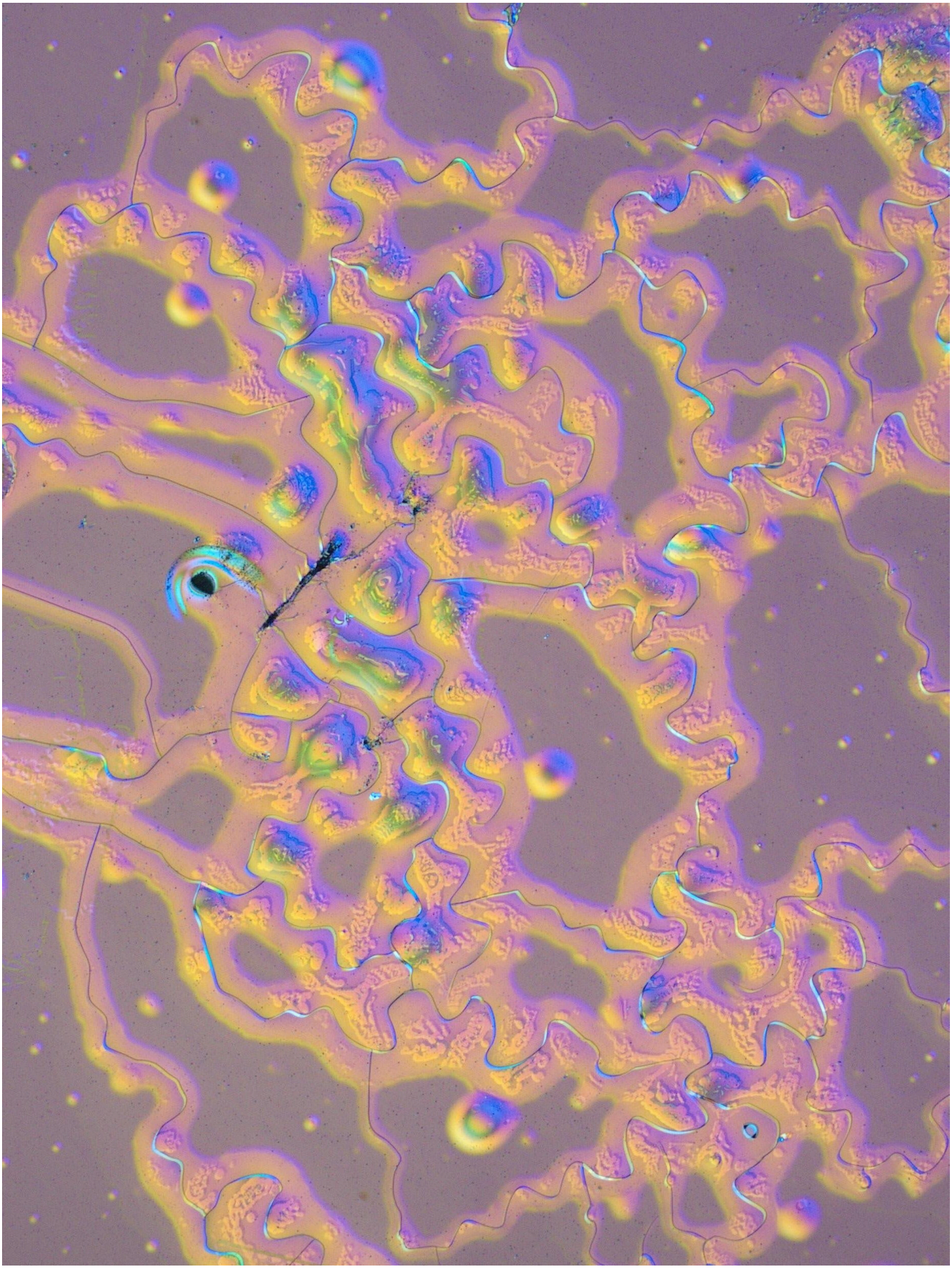


7 Years of Bad Luck

300µm

Aluminium oxide and Silicon carbide are never happy to be stacked together, and adhesion between those layers tends to be weak. Here a silcion carbide layer ended up fracturing into many pieces when the thin aluminium oxide ended up getting partially etched in a long hydrofluoric acid vapor process. The differential interference contrast image shows interference rings where the silicon carbide is bowing upward, no longer confined to the surface by the aluminium oxide.

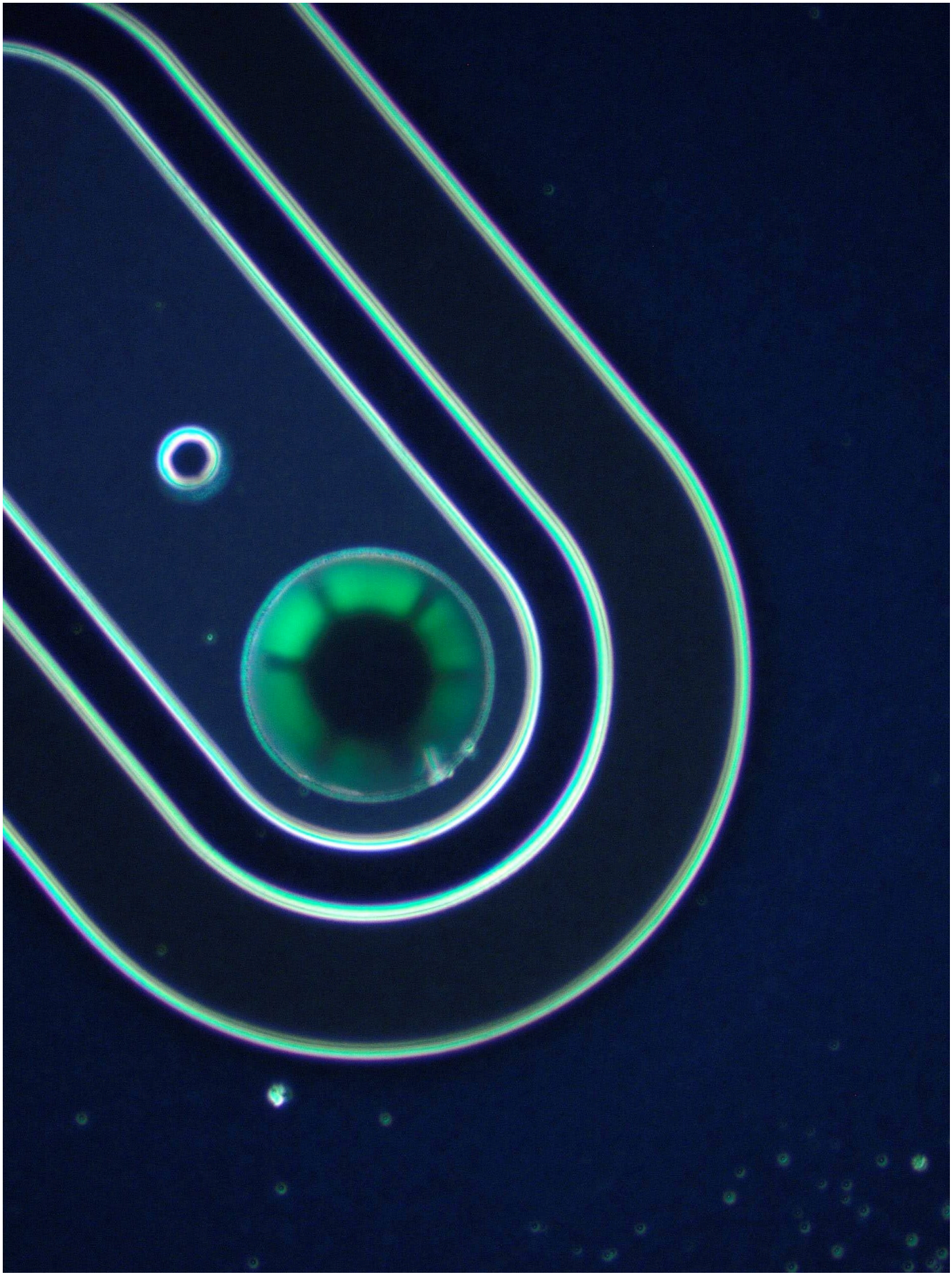
Olympus BX51



Another 7 Years of Bad Luck

100µm

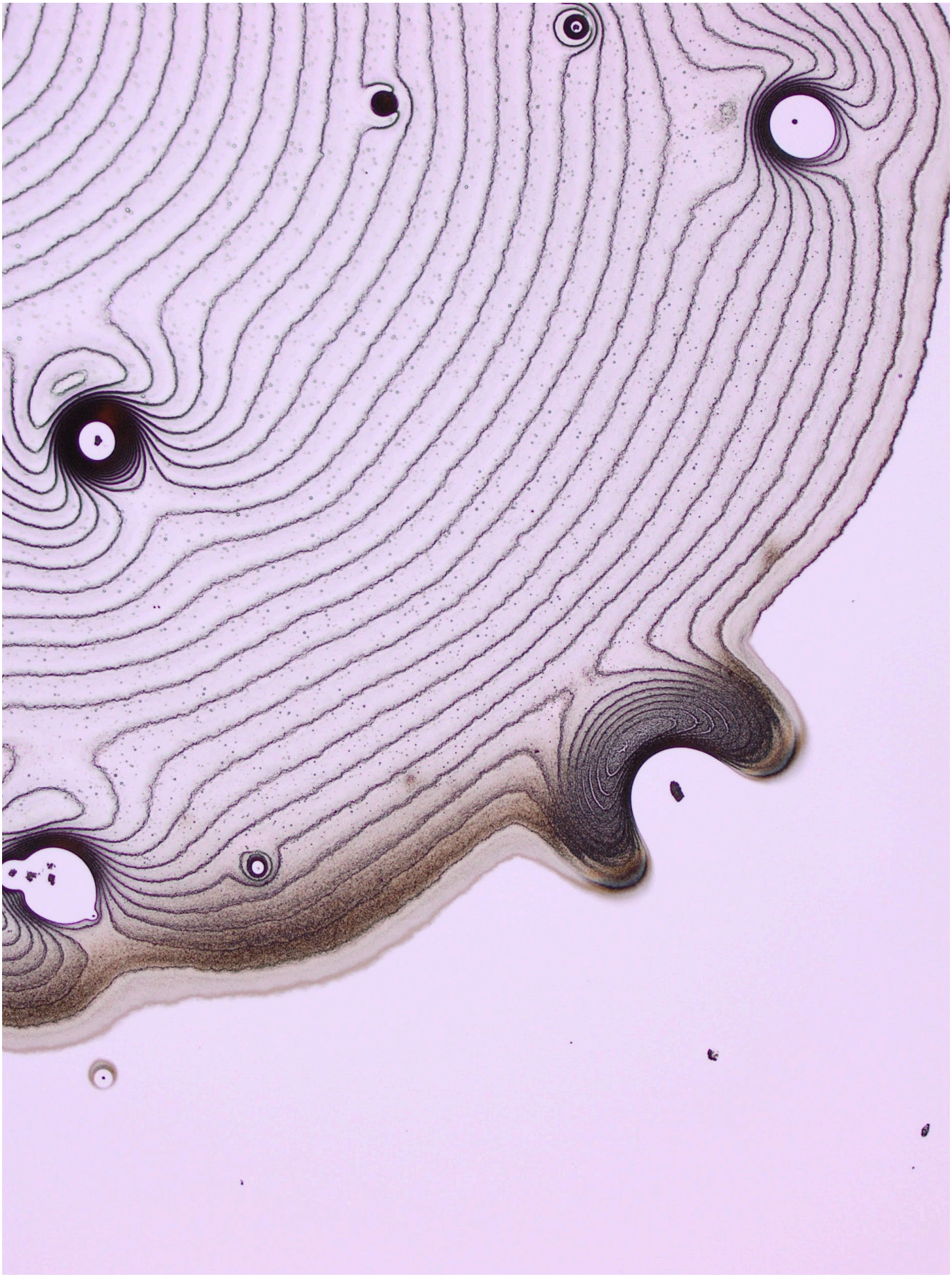
In this differential interference contrast image, the silicon carbide layer ended up cracking in many places, this time because the supporting silicon dioxide was partially removed by hydrofluoric acid vapor. In this context, formation of a crack in the carbide can be self-propagating, as the crack will let in more vapor, resulting in more support material removal, which will then result in yet more cracks forming. I think many years of bad luck await me at this point.



When the Substrate Stares Back

100µm

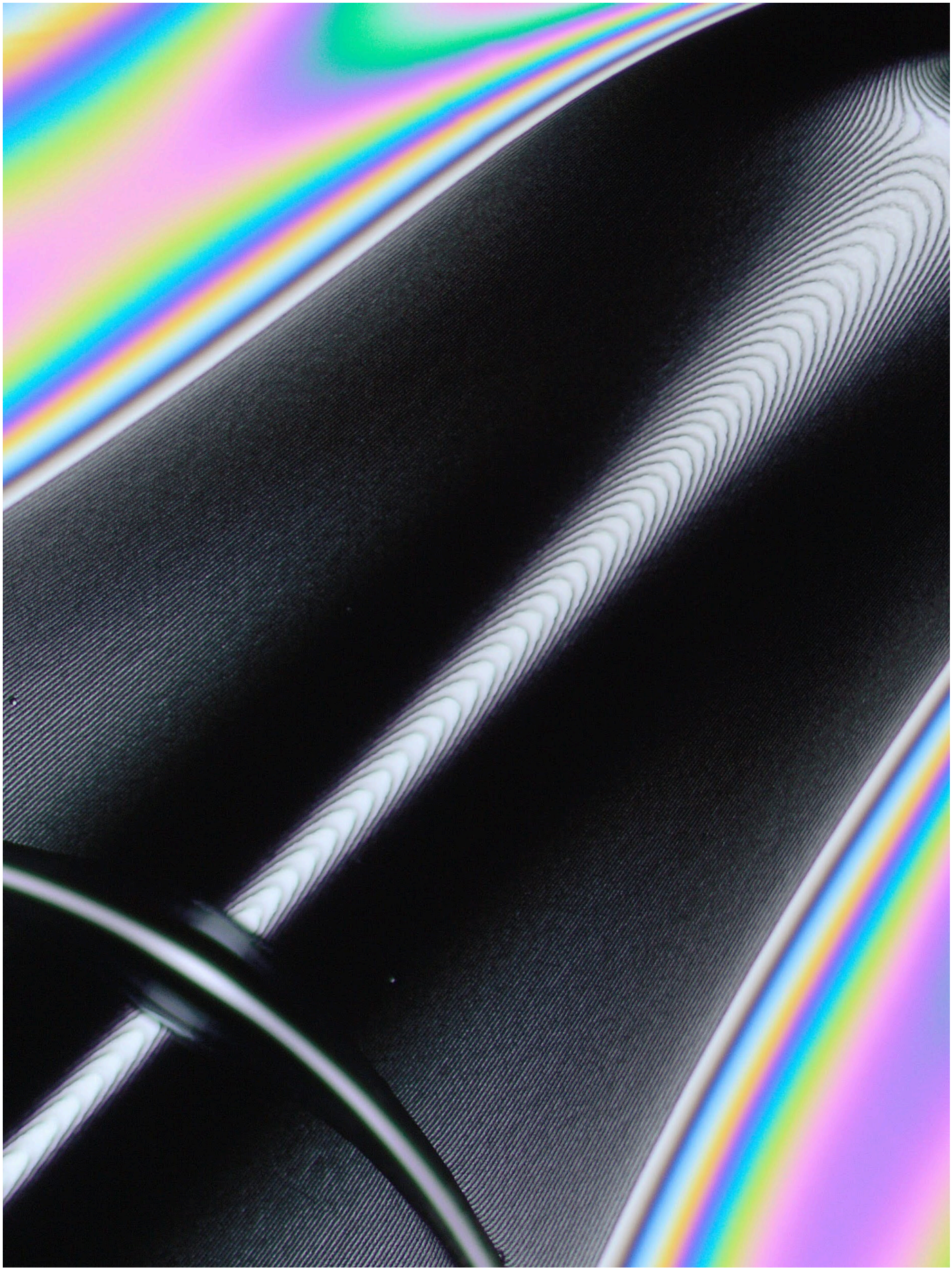
This suspiciously-eyeball looking shape is a formed image of the darkfield light stop of the microscope. I'm not enturely sure why this ended up being visible in this context; maybe there was a bit of lensing action happening in the through-hole etched into the silicon. It is always a good day when there is something of interest to see that does not involve a failed fabrication process.



Micro Terraces

300µm

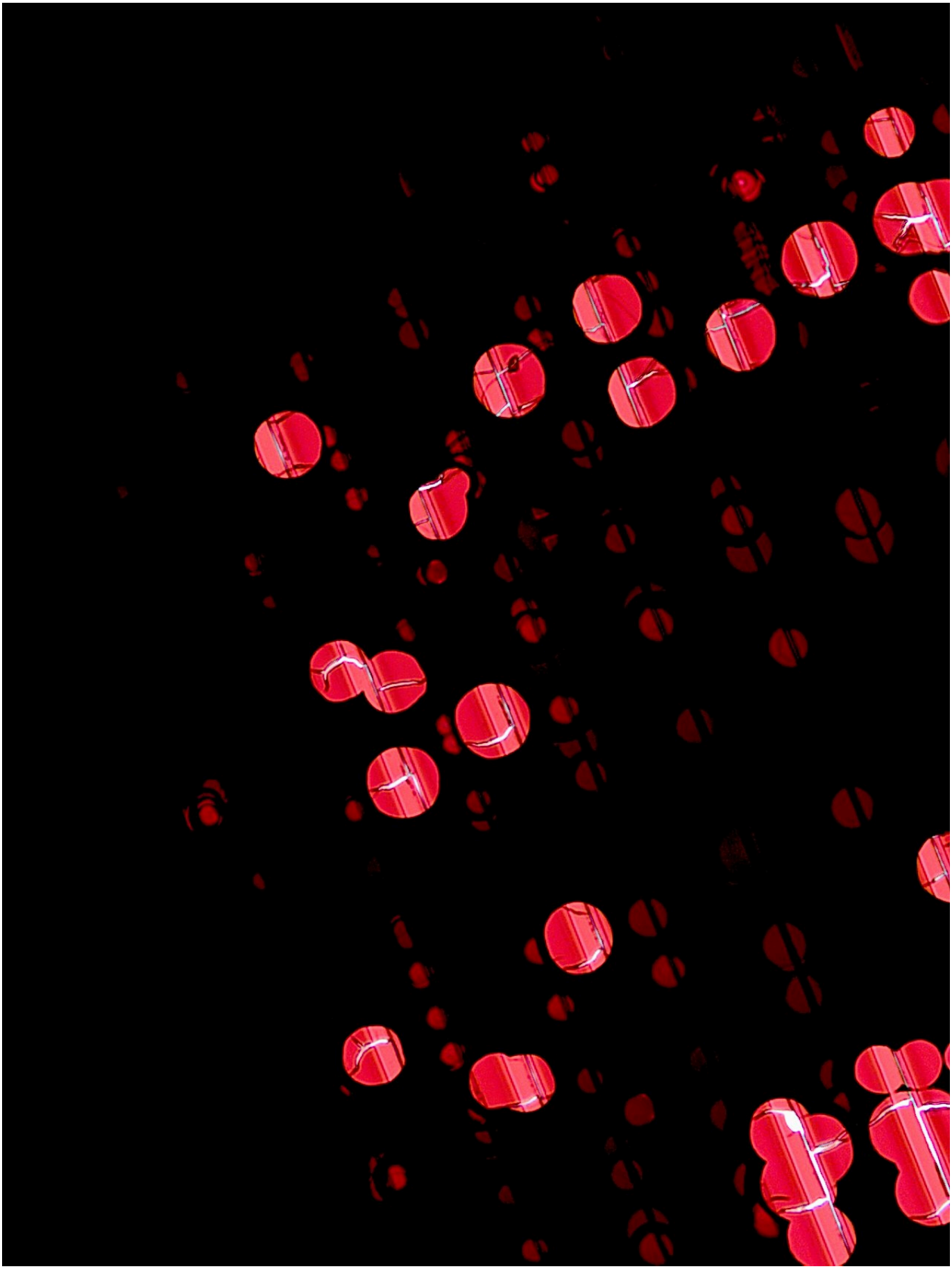
Here I found out the limit of a specific photoresist in masking a silicon etch process. When the photoresist mask is slowly eroded in the etch process, it will at some point start exposing the silicon to the etchant, resulting in unexpected patches of etched silicon. The silicon ends up etched in distinct terraces due to the pulsed nature of silicon etch process—it alternates periods of silicon etching and silicon passivation.



Micro Terraces II

300µm

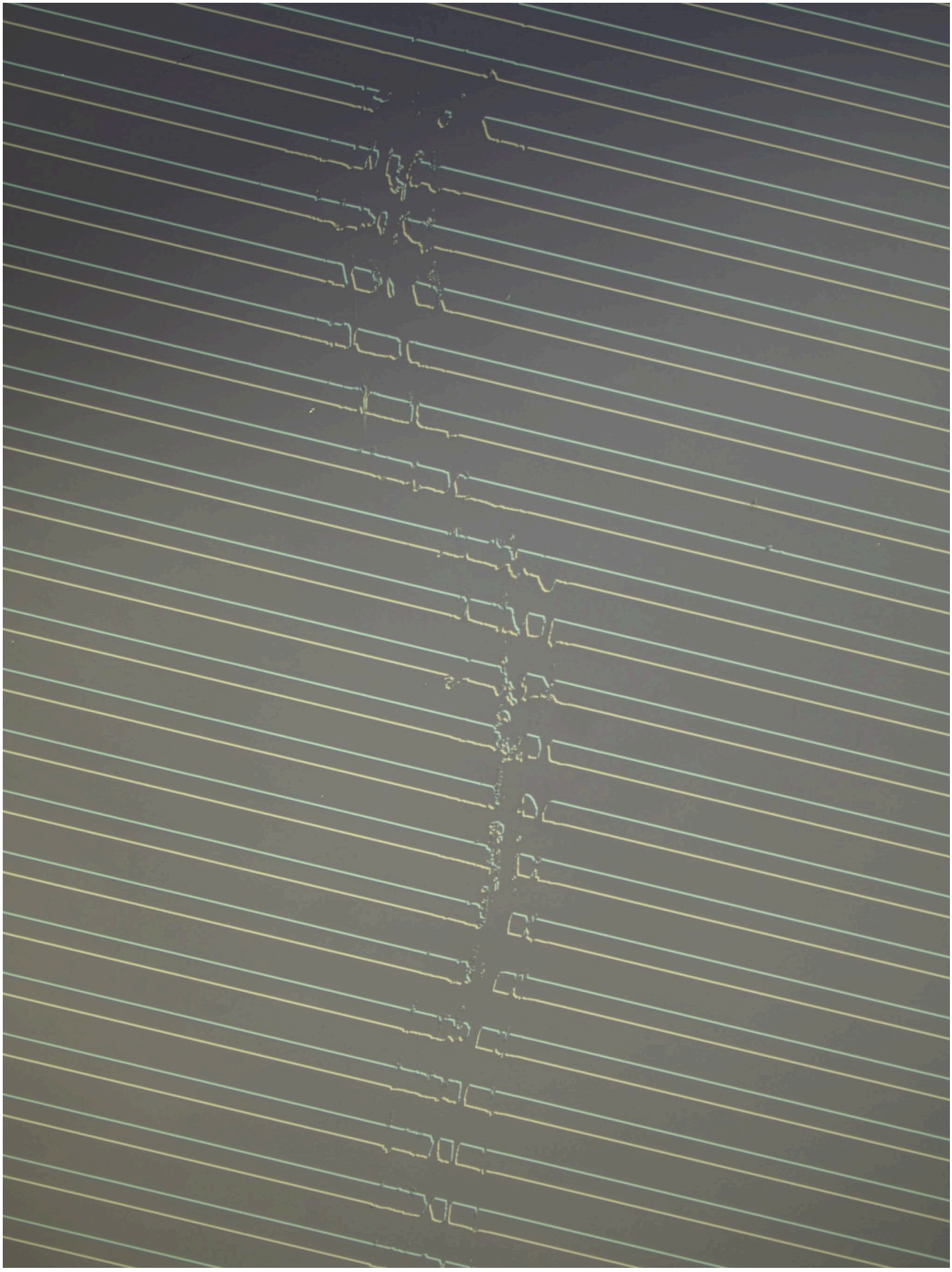
Another example of terraces etched into silicon when a photomask ended up eroding too much during the etch process. Here the colourful masking photoresist has not yet been removed. I wonder if this kind of pattern could be paired with grayscale photoresist to make topographical maps at the microscale. Anybody out there that would like to fund me to find out?



Lanterns

100µm

Here we are looking through the silicon substrate from the backside. Most of the silicon has been removed in a deep reactive ion etch process—but not all. The remaining few micrometer of silicon results in transmitted light having a red cast, as silicon has lower light absorption in the longer (more red) wavelengths. The strange hole pattern was due the etching machine conducting a 'spontaneous wafer dicing' operation. It turns out that breaking your substrate into many pieces in the machine is not a recipe for success.



Tweezer Discipline

100µm

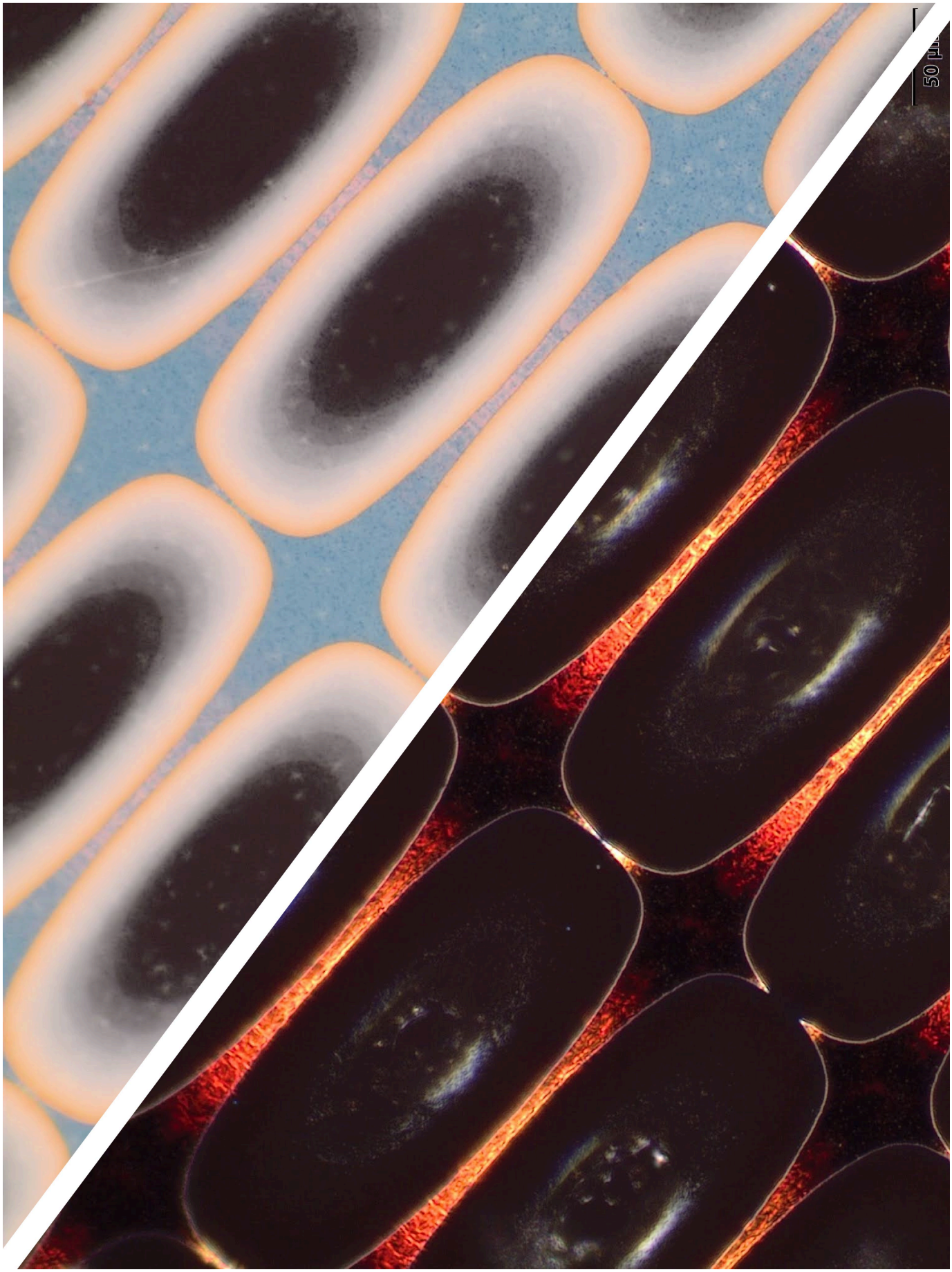
These structures were ion milled directly into a standard microscope coverglass. This would allow for easy integration into microscopy workflows. We intended to grow protein filaments known as *microtubule*s from these lines visible in the image. In this particular area of the substrate, somebody got a little careless with their tweezer and scratched the masking photoresist. I fear we will never be able to identify the one responsible.



Broken Stencil Membrane

150µm

This poor silicon nitride membrane ended up breaking during the fabrication process. One aspect of the membrane that probably contributed to the collapse was the fact that it was full of small holes that related to its function as a stencil mask. In fact, this kind of large suspended holey membrane was used to produce the gold patterns as shown in Inorganic 12. A large suspended membrane area and many potential weak points is a dangerous combination.



A Fool's Hope

150µm

When looking back at your earliest experiments, you may start to question your decisionmaking. This is one of those examples. Here a silicon wafer was etched using deep reactive ion etching and a silicon nitride stop layer. The etch rate of silicon nitride is signifcant (see also Inorganic 15), so this etch process would produce a gradient of different silicon nitride thicknesses—and a hole in the middle. Somehow, the hope was that graphene present on the silicon nitride may remain in some areas. Later ideas were much better.

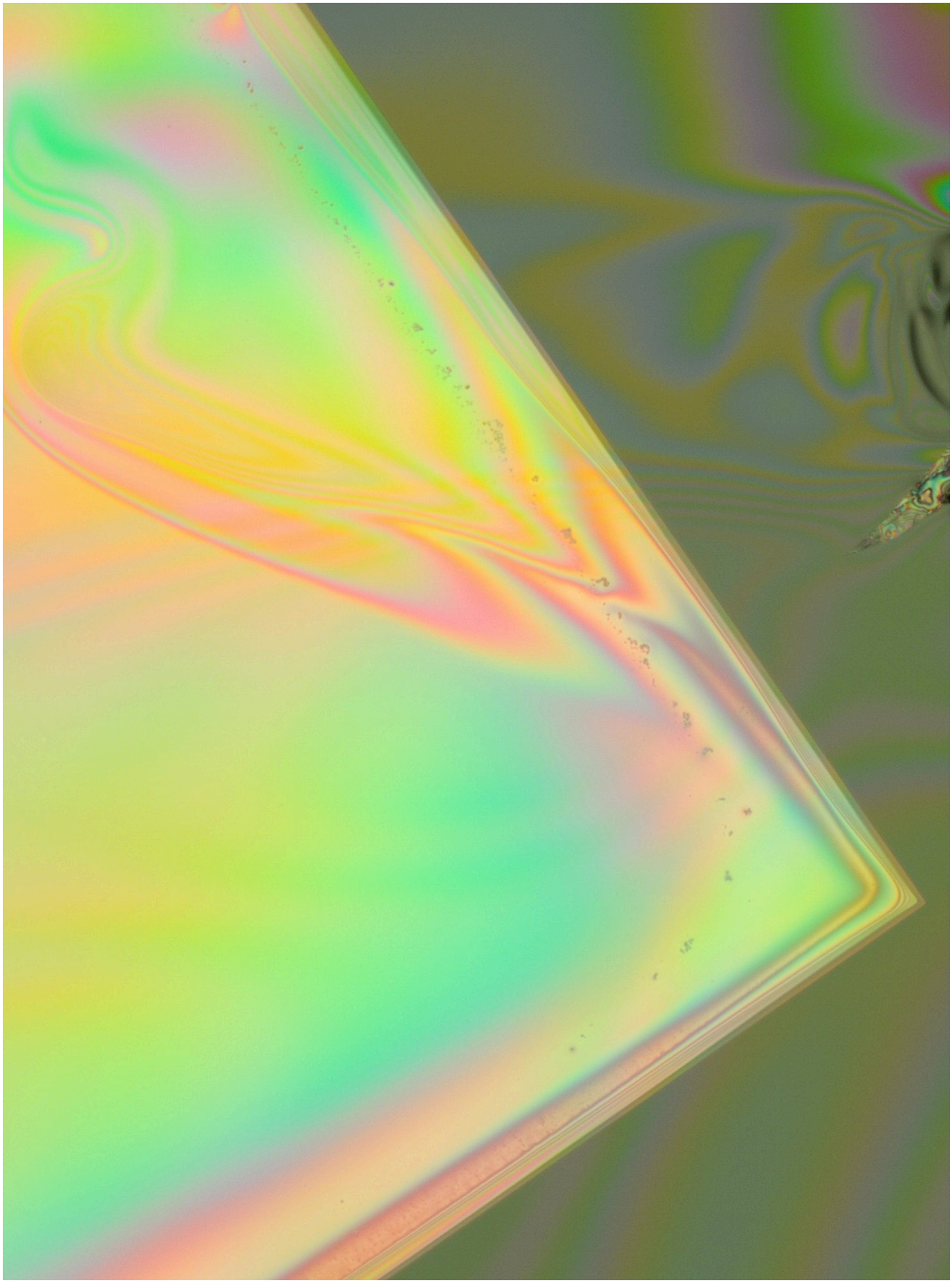
Olymypus BX51

2. Polymers

Organic compounds have properties that usually vary significantly from inorganic materials. They are usually soluble in compounds that inorganic materials are entirely insoluble in, and sometimes quite resistant to chemicals that rapidly etch inorganic compounds. These properties are essential in the function of photoresist layers, which are central to all patterning steps, as each of the many 2.5D layers that are etched (or otherwise patterened) on typical devices are first masked with a (photo)resist coating. A patterned resist layer can effectively mask the inorganic layer below, protecting areas with resist present from being removed, and thereby enabling site-selective removal of inorganic material.

Photoresists find such broad application because they themselves can be patterned with UV light. The spincoated polymeric layer will, depending on the type of resist, either undergo (polymer) chain scission in illuminated areas (through an intermediate photosensitising compound) or become more crosslinked. Resists of the former type are know as *positive* resists, while the latter are referred to as *negative* resists. The light exposure itself changes the composition of the resist layer, but becomes only apparent in the chemical development step which is carried out after exposure. During development, either the exposed or only the exposed parts dissolve into the developer solution. In either case, the result is the same: a patterned polymeric layer whose shape is a copy of the light pattern that was used for exposure. Lithography, but with light instead of a pencil – photolithography.

Besides their differences in chemical solubility, the thermal resistance of polymeric materials like photoresist is also much lower than that of organic compounds. At temperatures above their glass transition temperature, resists will start to behave like a liquid; at temperatures above that they will start to become increasingly crosslinked, and at temperatures higher still they will thermally decompose. These crosslinked resist will then only be partially soluble in typical removal solutions, which can in turn lead to some surprising microscopes (and a failed device). As these polymeric layers can also release gas when heated, they have the tendency to form 'blisters' when covered by an impermeable layer. As gas is released with nowhere to go, these blisters can take a large range of shapes and sizes depending on the experimental conditions.

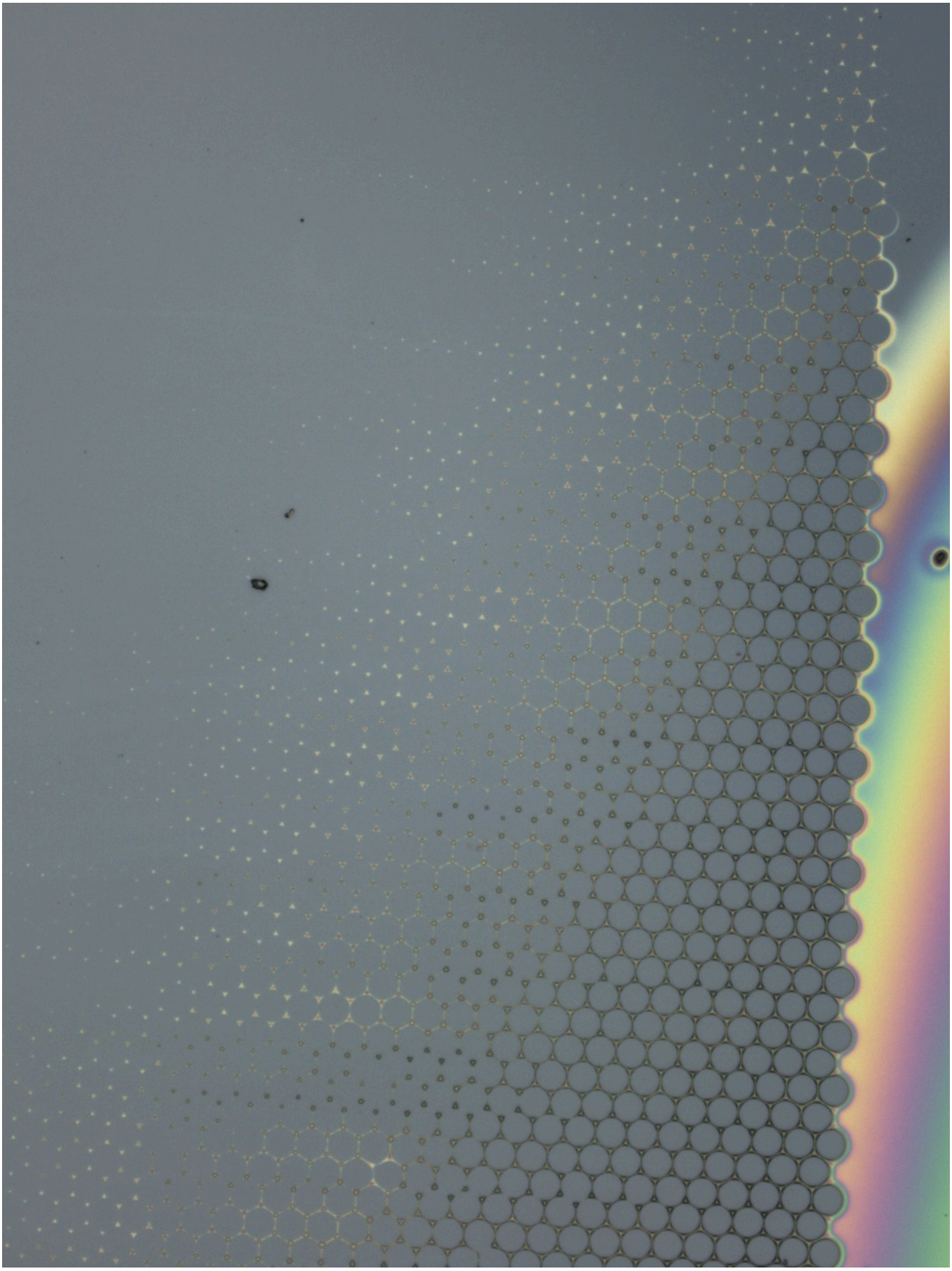


Liquid Rainbow

25µm

Photoresist will, by virtue of the same thin-film interference principle that gives colour to inorganic layers, produce colourful interference when present on a surface. The range of colours by a single coating is often much greater than for inorganic layers, as the photoresist is spincoated onto the substrate in liquid form. Proximity to the substrate edge, and local topology such as etched features in the silicon nitride as in this image can locally increase the resist thickness, and thereby alter the interference colours.

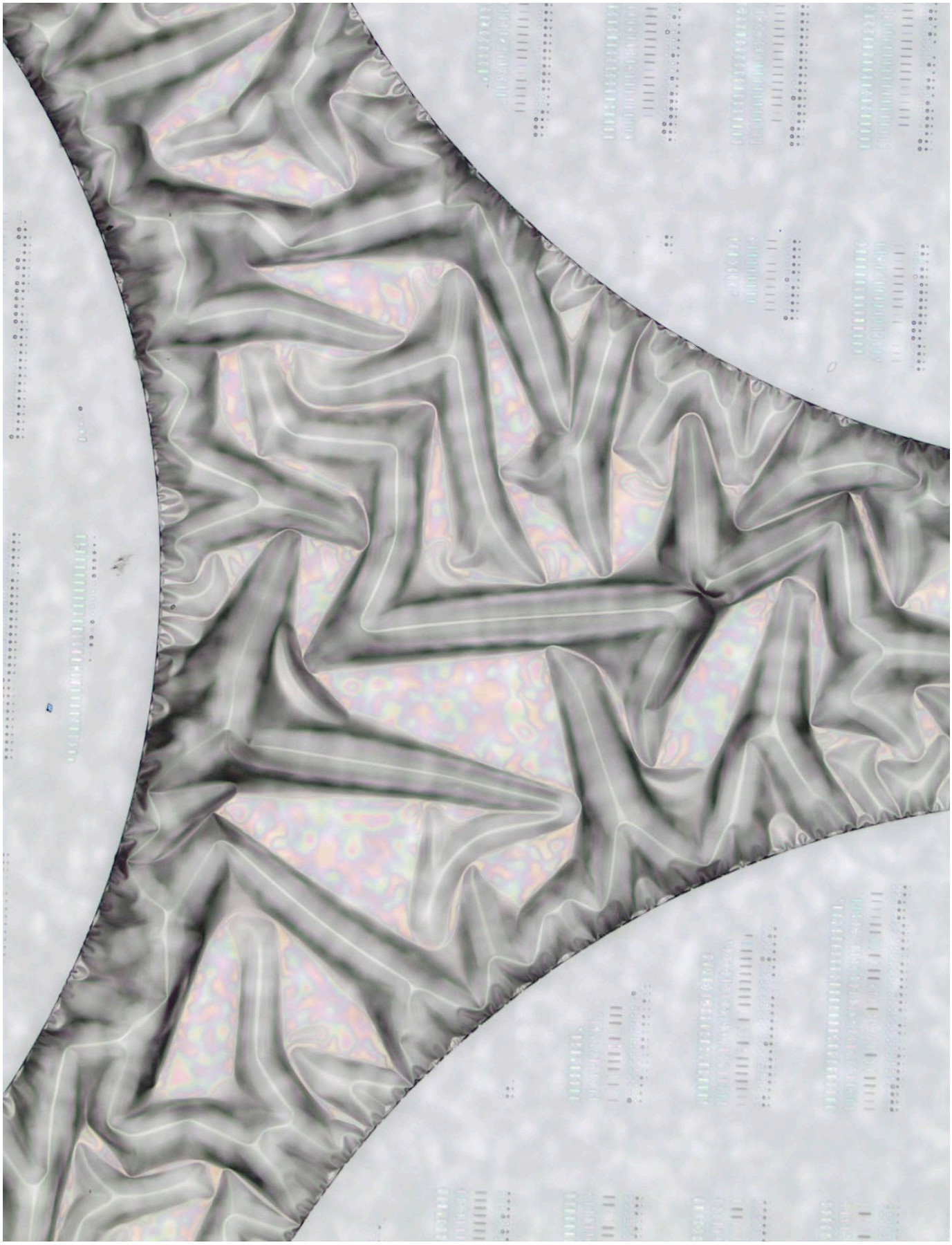
Olympus BX51



Circles to Hegaxons

200µm

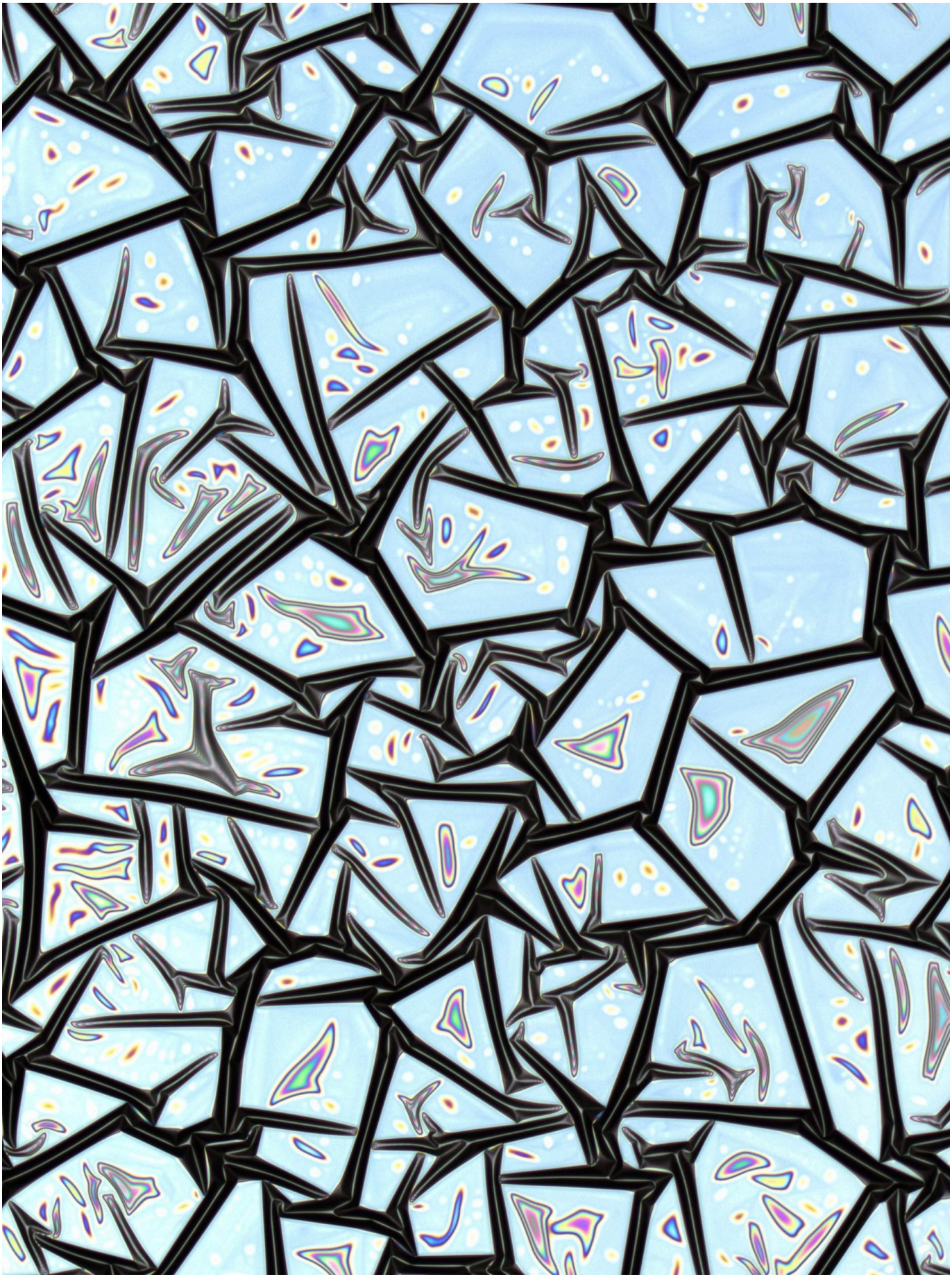
This image shows a photoresist pattern that is in the process of being ashed by an oxygen plasma process; a common approach to remove organic residue from a substrate surface. The pattern present on the wafer was a triangular lattice of circular holes. As the rate of plasma etching varies across the surface, a gradient of 'material removal' can be seen. On the right, where little removal has taken place, the circles remain. On the left, the pattern turns into a hexagonal lattice of triangular shapes.



Holding On

200µm

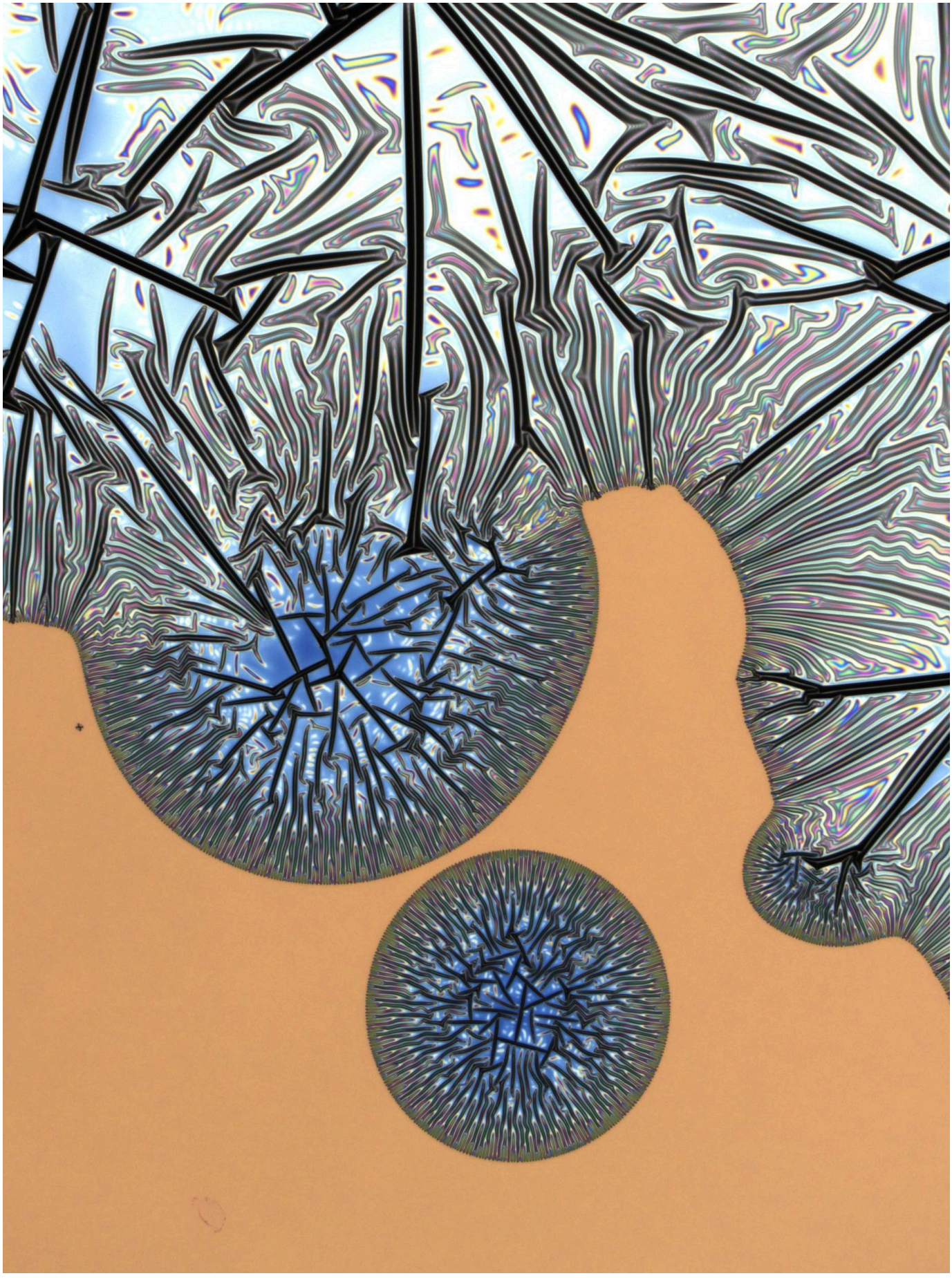
In this device, we wanted to make some structures on glass that could be transferred to a TEM grid. Rather than etching the glass in hydrofluoric acid or using a plasma process, a lift-off process was attempted. The glass is deposited onto a substrate containing patterned polymer (photoresist). After deposition, the polymer is removed, and—hopefully—the glass comes with it. In this instance, the glass was not ready to leave the substrate, and ended up staying behind as a wrinkled layer.



Carbide Wrinkles

300µm

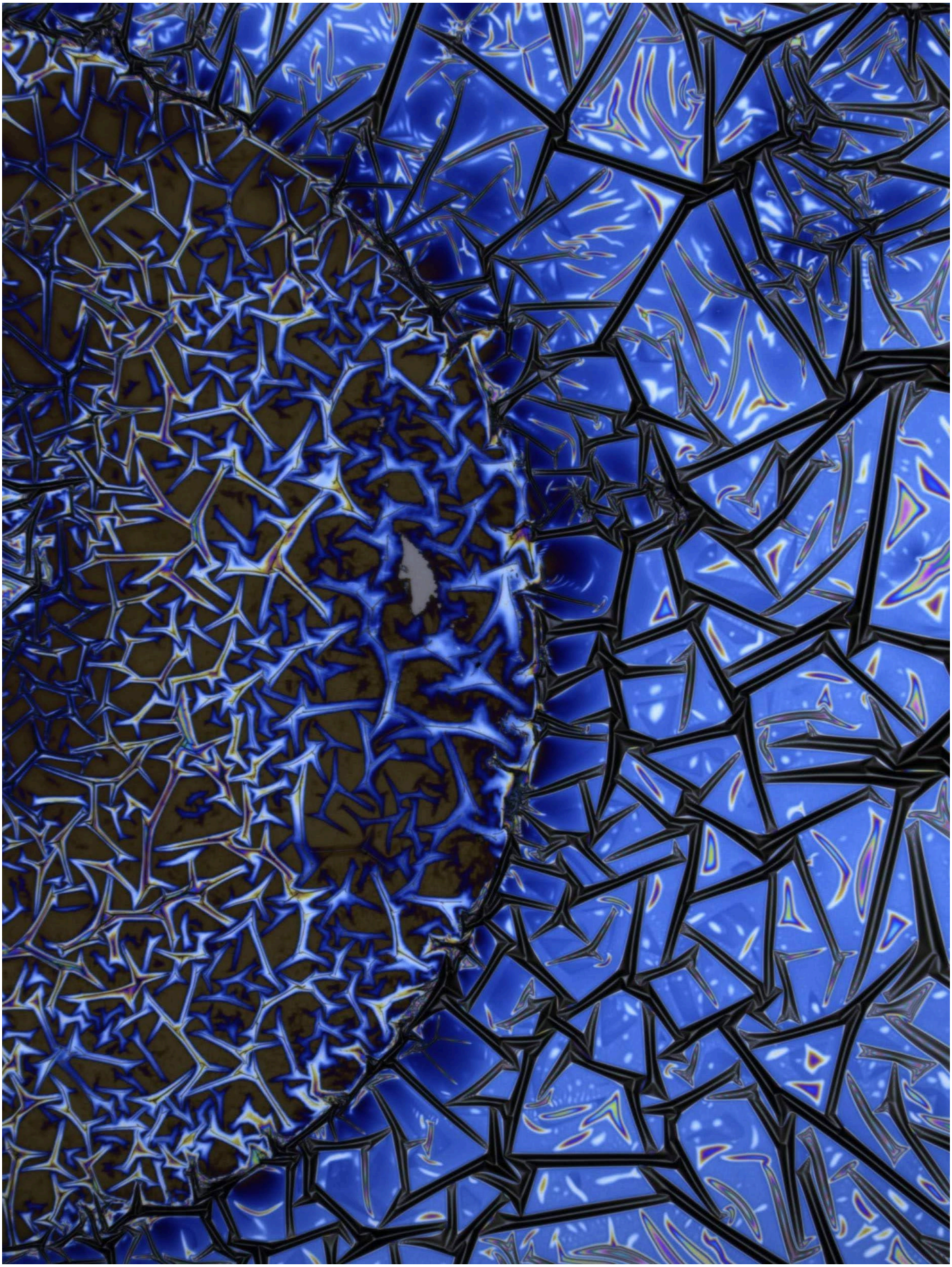
In this instance, we are looking at an area of the wafer that did not contain any pattern; a 40nm silicon carbide layer was originally supported by a photoresist layer, which was removed in organic solvent. The silicon carbide ended up in this aesthetic wrinkled pattern as it re-adhered to the surface. Some of the coloured sections might be due to remaining solvent trapped under the silicon carbide layer.



Carbide Wrinkles II

300µm

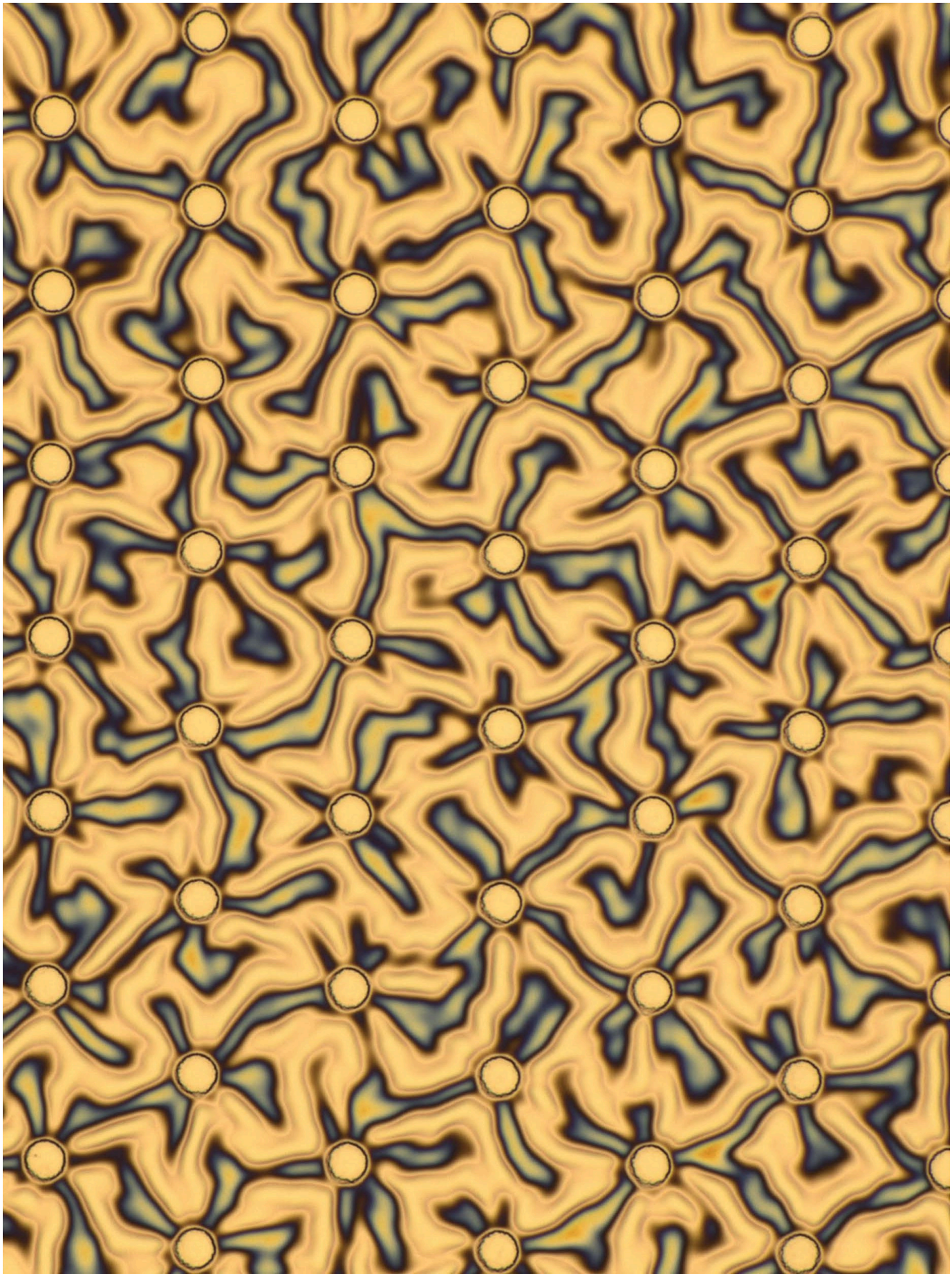
This is the same sample as Polymers 04, but now further away from the substrate edge. if the silicon carbide layer is free of any defects, the solvent has no way to reach and remove the supporting polymer. Because we are further away from the substrate edge (where the solvent can always creep underneath the silcion carbide), not yet all the polymer has been removed. We see some sections that are just beginning to be dissolved in solvent, and areas where the photoresist is still solid.



The Carbide Corral

300µm

The third and final installment of the silicon cabride wrinkles shows a change in wrinkle texture. Maybe the circular section contained a pinhole, allowing removal of the supporting photoresist before the bulk of the resist was removed through other openings. I think the brown coloured section of the circular corral represents the silicon carbide being fully in contact with the underlying substrate with no trapped solvent or photoresist. Or maybe the micro-cattle ate all the vegetation there. You can choose which one you believe.

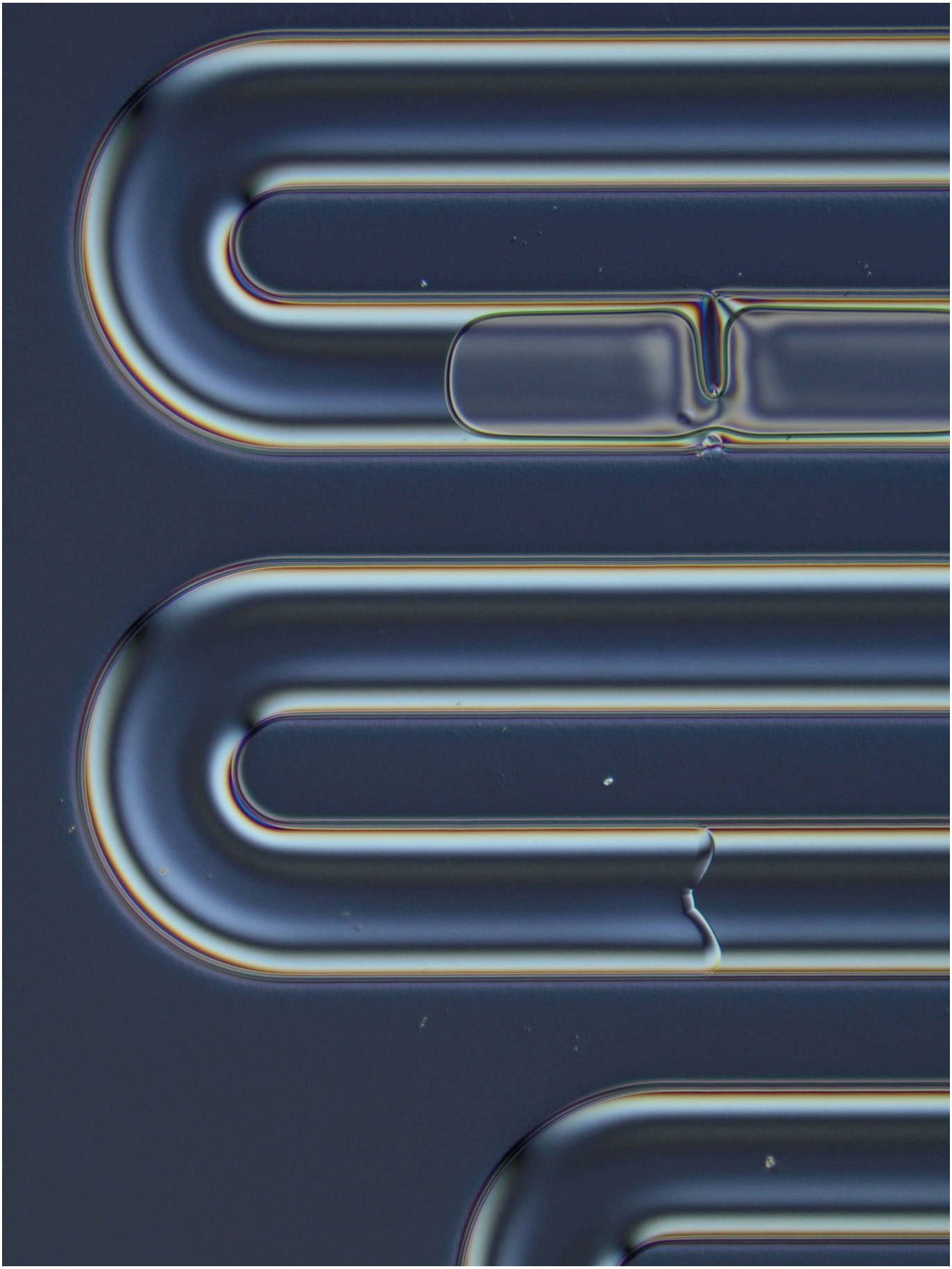


Field of Flowers

50µm

One of the big challenges of working with polymers is the limited thermal budget. Here, a photoresist layer was coated in an amorphous silicon layer using a—what was supposed to be—low temperature deposition process. The organic layer clearly disagreed, becoming strongly deformed in the process. The regular triangular lattice pattern of holes was developed into the resist before deposition—with the intention of increasing the strength of the deposited silicon layer. Too bad the photoresist had other plans.

Olympus BX51

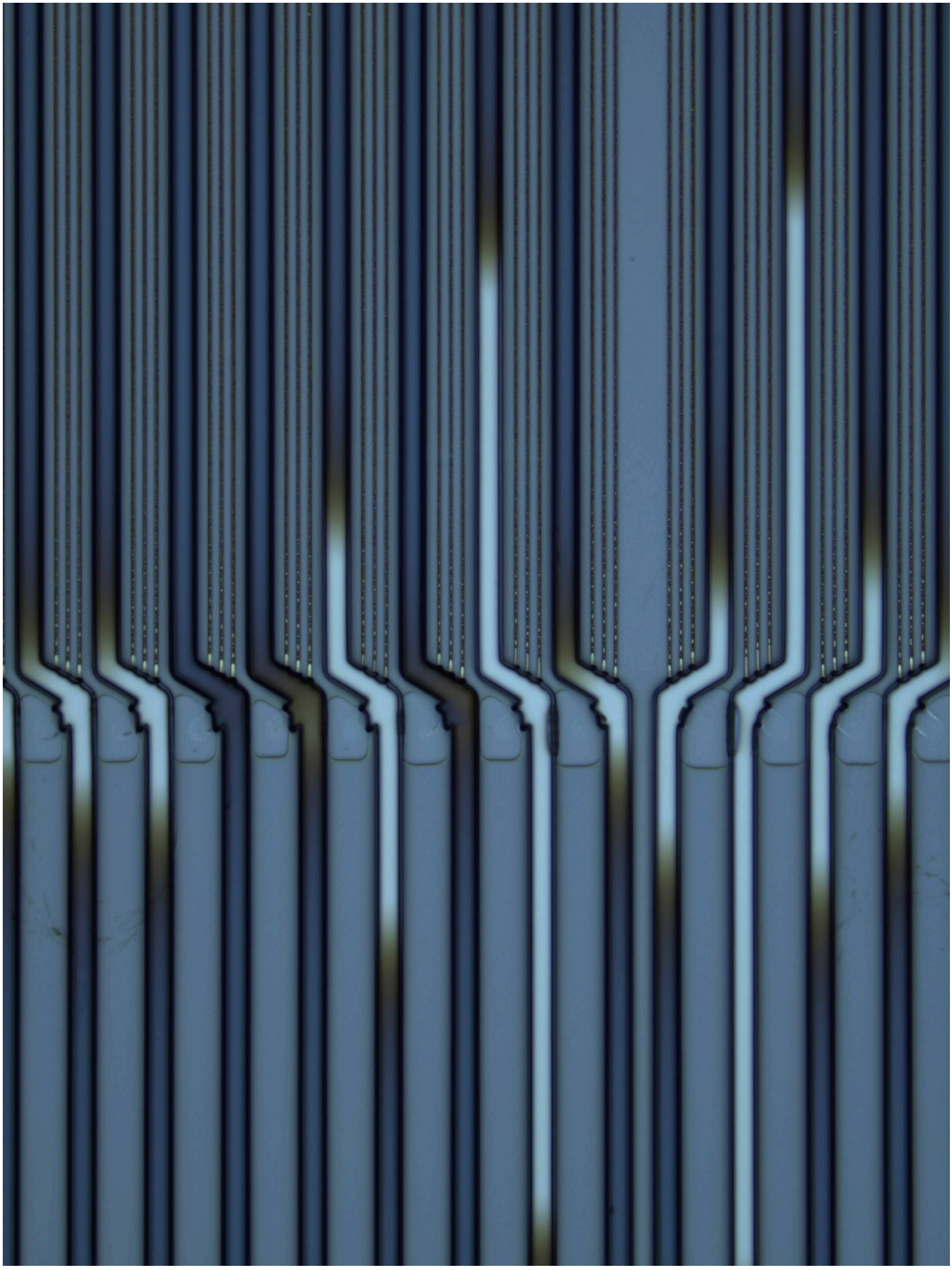


Somebody Call a Plumber

100µm

One of the core challenges of my project involved making microchannels with a sacrificial polymer layer. Here we see one of the test pieces, where a microchannel snakes along the screen—still mostly filled with organic solvent. A lone air bubble is trapped in an (accidental) constriction in the channel. Further down in this differential interference contrast image we see a crack that has formed in the microchannel. Two big flow problems in a single field of view — time to call a plumber.

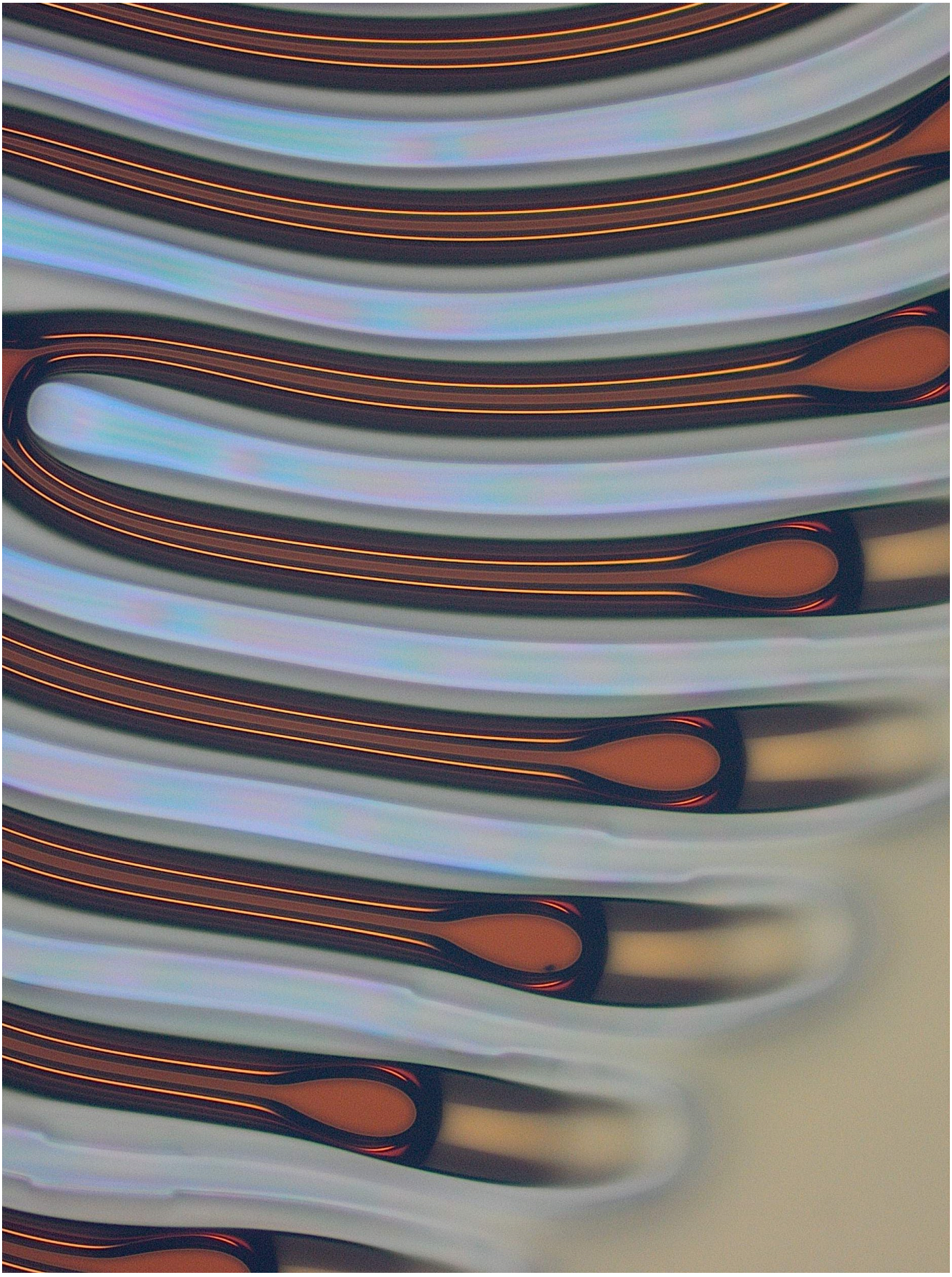
Olympus BX51



Carbon Patina

100µm

In this older microchannel and nanochannel arrangement, a little bit of residual polymer was left behind inside the channels. In this instance, I was hopeful that we could ignore that problem, and hope for the best that some of the channels would remain usable. After a high temperature and high power plasma deposition of silicon dioxide, it appears that whatever residual polymer was inside the channels had turned into ash and coated almost the entire inner walls of all the channels.

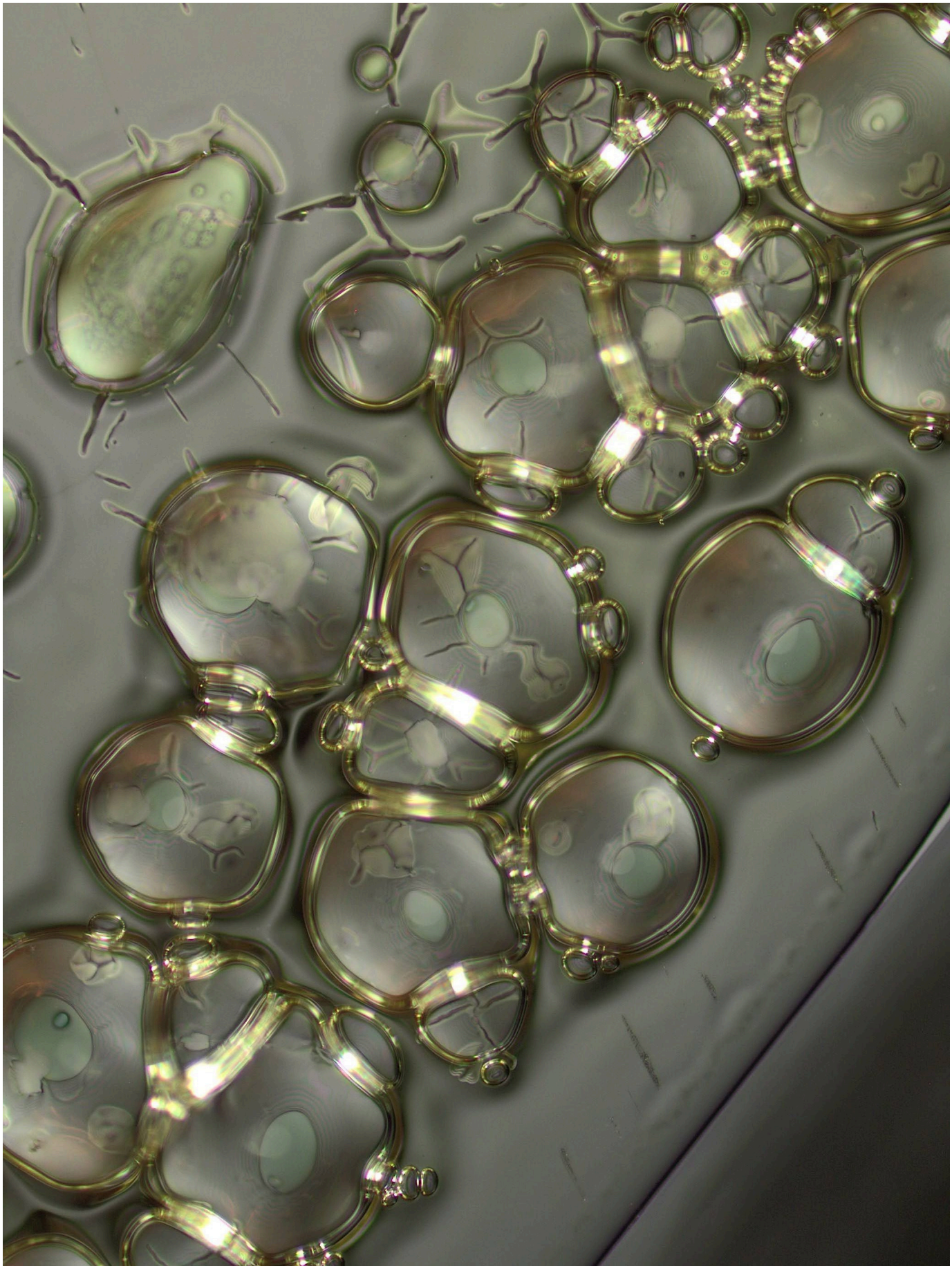


Micro-Coral Polyps

200µm

The variety of strange and otherwordly structures you can find when you heat up a photoresist layer beyond reasonable temperatures is endless. Here, photoresist was used as a makeshift thermal interface layer for a deep reactive ion etching process. This resist layer was heated to high temperatures, with nowhere for any gas present in the layer to escape. What exactly is going on in this area is unclear to me, but I think we can just enjoy it for what it is.

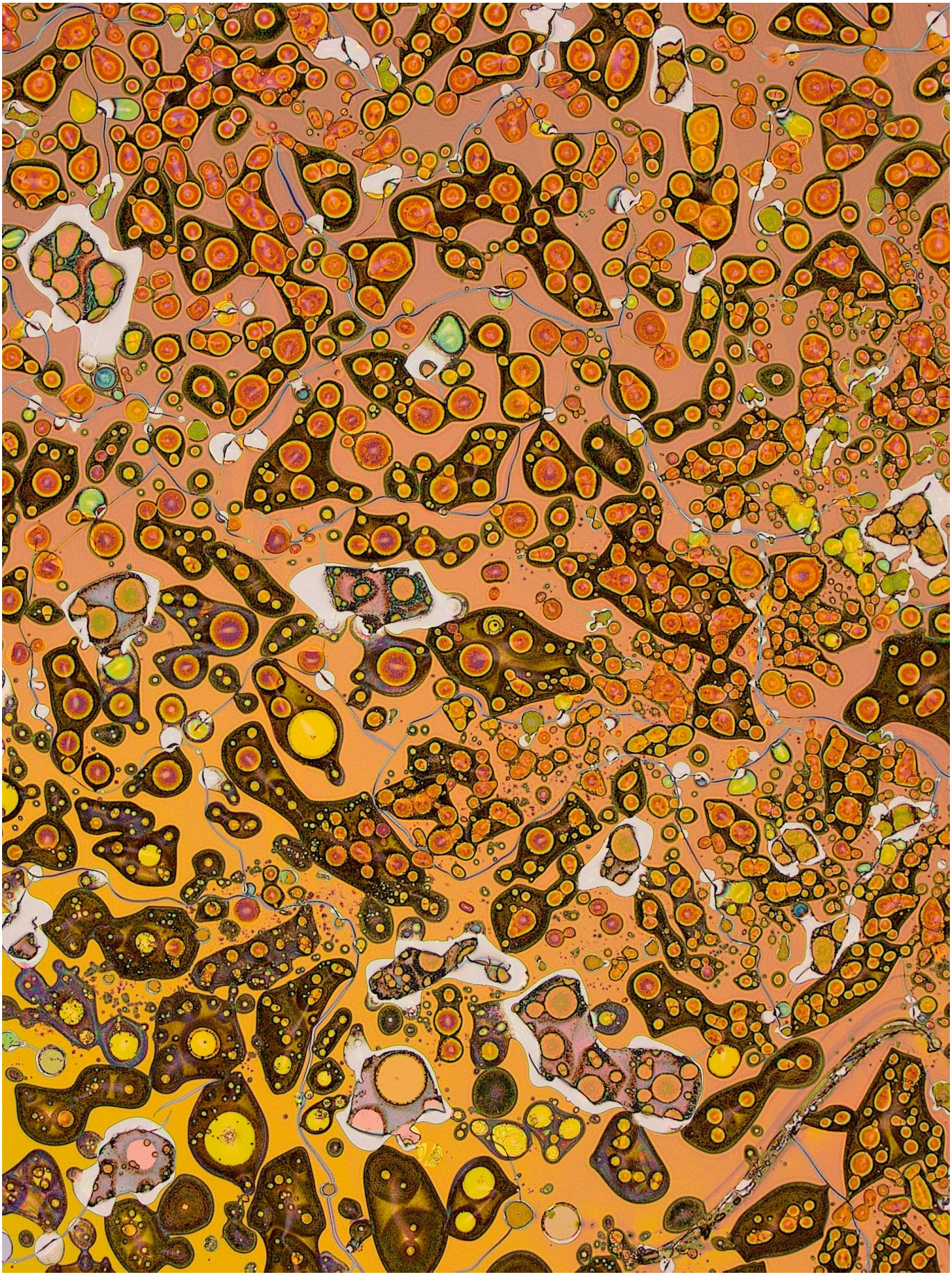
Olympus BX51



Manifestations of Plague

200µm

Hydrofluoric acid (HF) is a rather scary compound, being extremely toxic by being able to easily penetrate through the skin. There, it sequesters calcium and thereby intereferes with the functioning of the nervous system. Unfortunately for us, it is also extremely useful in etching oxides such as glass. Here HF vapor was used to etch some glass on the substrate, with photoresist still present in other areas of the wafer. Photoresist is a very poor mask for HF vapor, as the gas can pass through it, and cause local delamination and formation of blisters.



The Blister Supreme

200µm

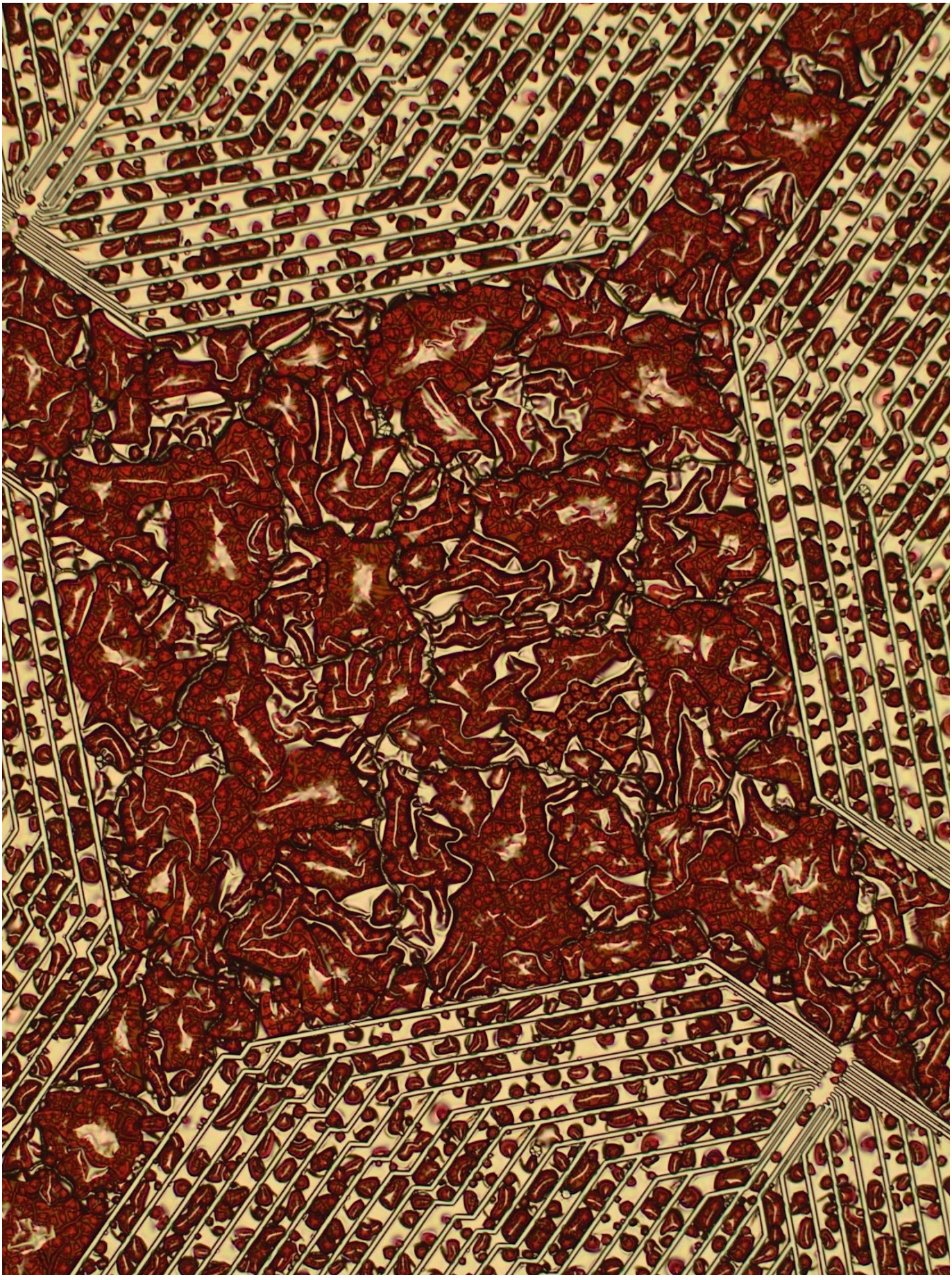
Another masterpiece by the famous artist “very hot plasma deposition process”. This time the photoresist layer was entombed by a thick silicon dioxide layer. With no pattern present in the photoresist prior to deposition, the plasma deposition machine has a clean canvas to work its magic on. I would say this is one of its more energetic pieces that highlights the dynamicity of the genre.



Still Life with Fruits

200µm

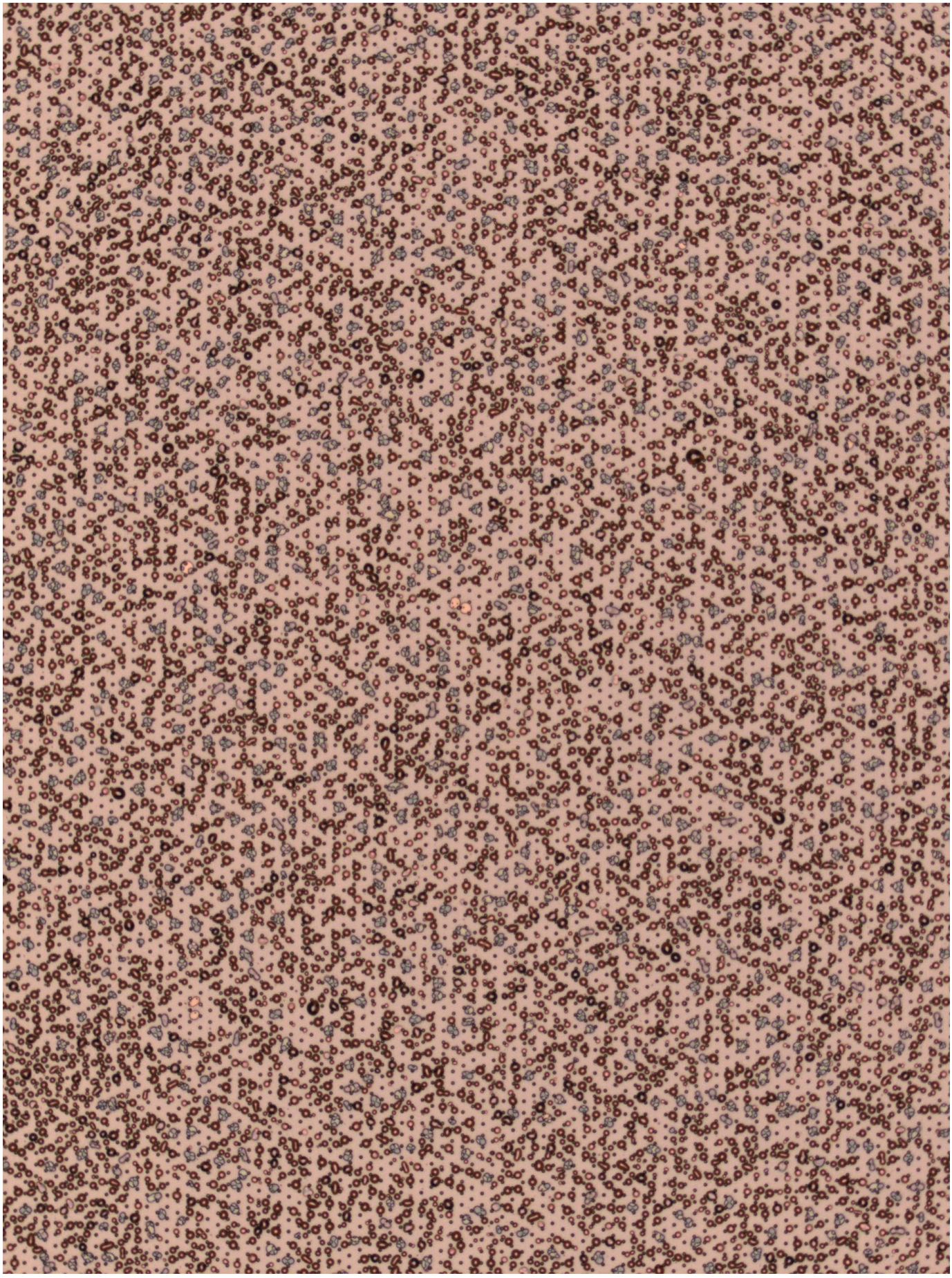
In this image, the setup is similar (photoresist + silicon dioxide), but the resulting patterns are quite different. The majority of the photoresist area appears entirely smooth, but then we find these radial patterns in spots on the wafer. This is likely an area where gas that slowly gets released from the photoresist layer ended up bulding up, and maybe even eventually breaking through the silicon dioxide layer.



Marmelade

200µm

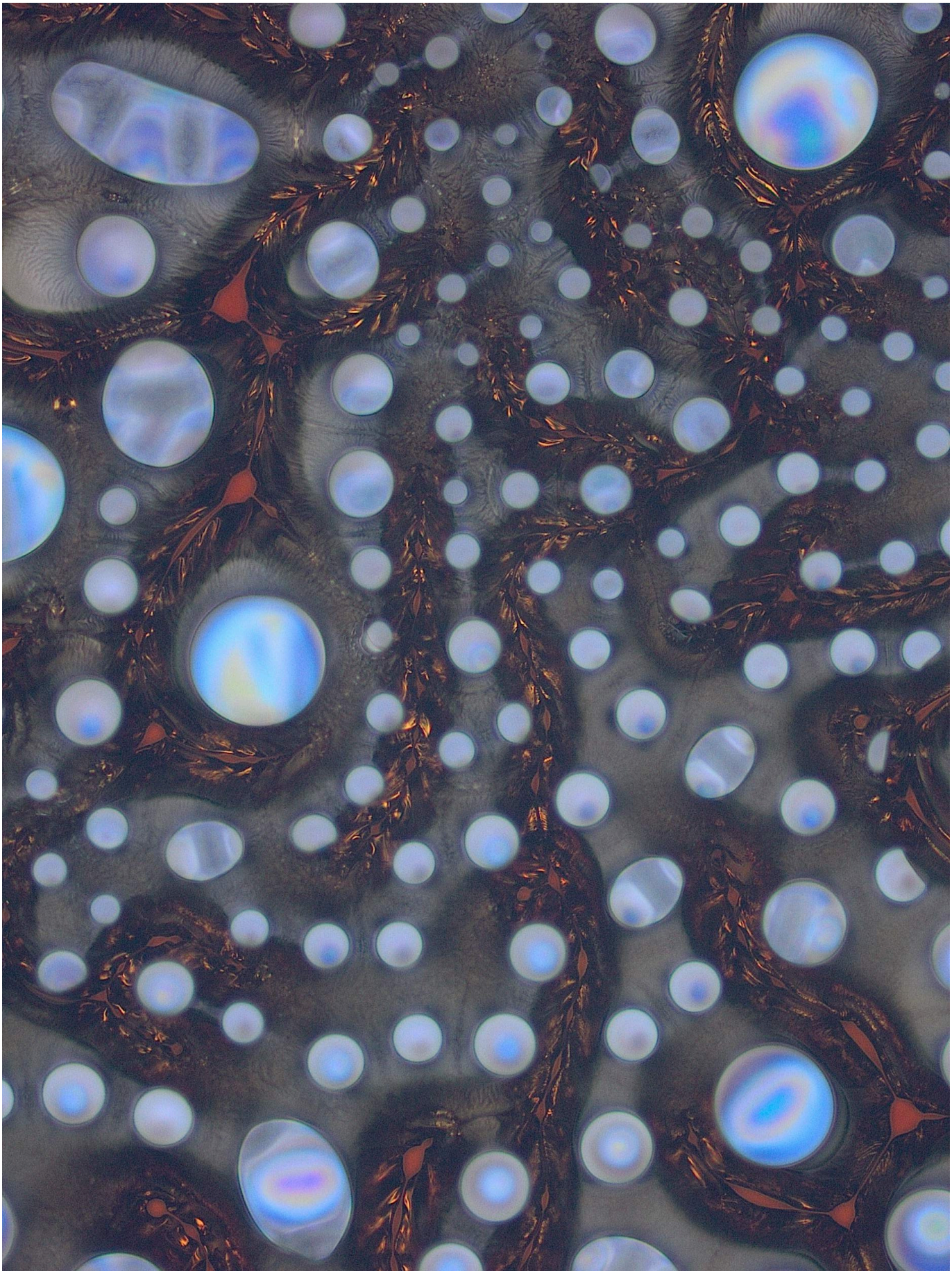
Yet another example of a plasma deposition process (this time of silicon carbide), that is too hot for a supporting polymer layer. In this example, the silicon carbide and partially delaminated resist produced a beautiful vibrant red colour. In areas where the resist managed to retain adhered to the substrate, a gold-like colour remained. An aside: all these images in sequence give the appearance that I am either unable to learn from my mistakes, or doing this deliberately. My lawyer told me not to respond to that if asked.



Random Number Generator

200µm

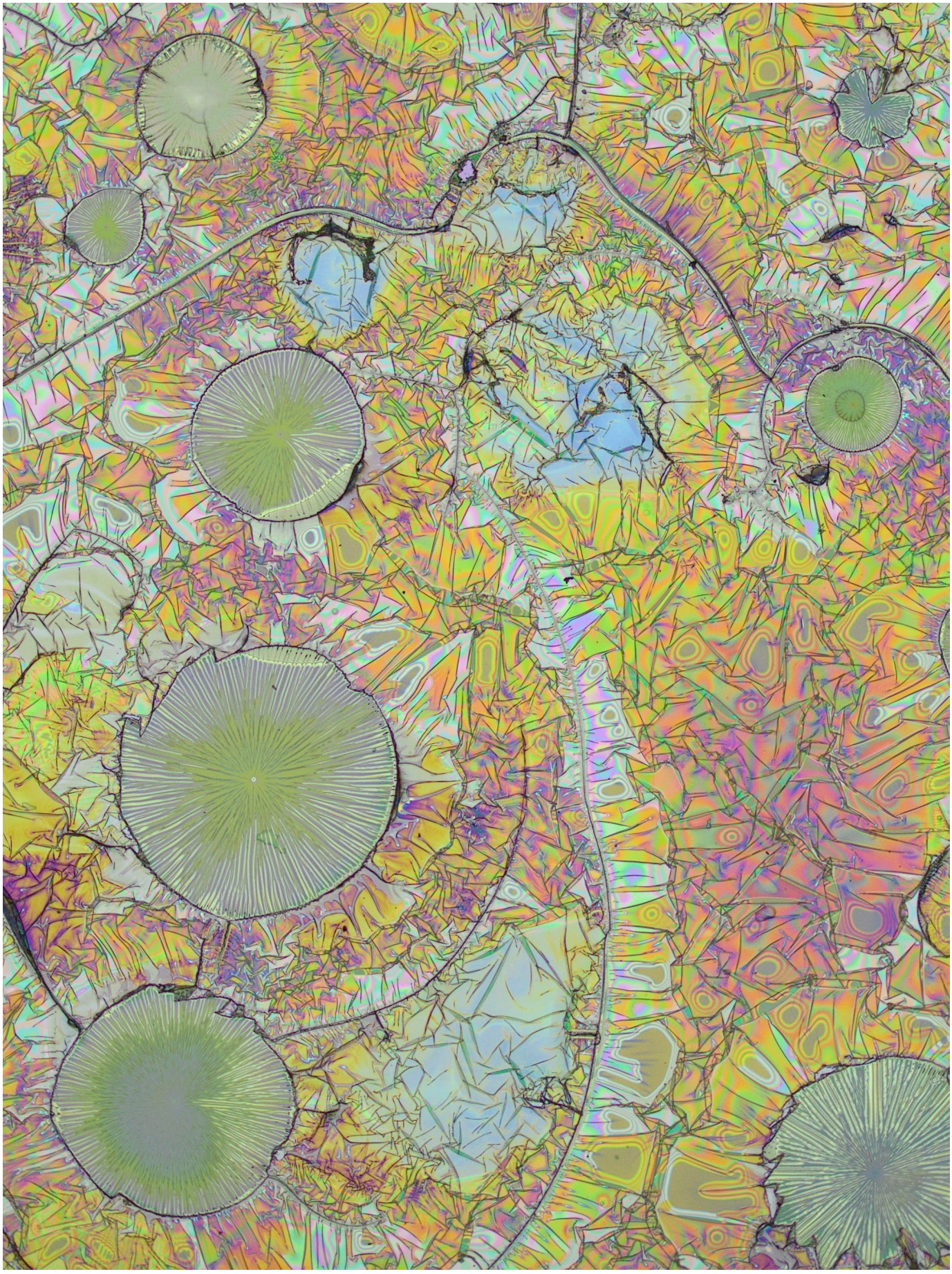
At this point the recipe is starting to get predictable: inorganic layer on polymer layer. Here, the densely spaced triangular arrangement of openings in the resist (just barely visible) prevented gas bubbles from pooling together, keeping delamination confined to the space between these openings. I think one could use this as an incredibly inefficient way to generate truly random numbers.



Slithering

300µm

This landscape is taken from the same wafer as Polymers 10. The line pattern in the resist reminds me of snake scales. Just goes to show you; you can put a man in a high tech microfabrication environment, but you can't get the biological snake-recognition response out of him.

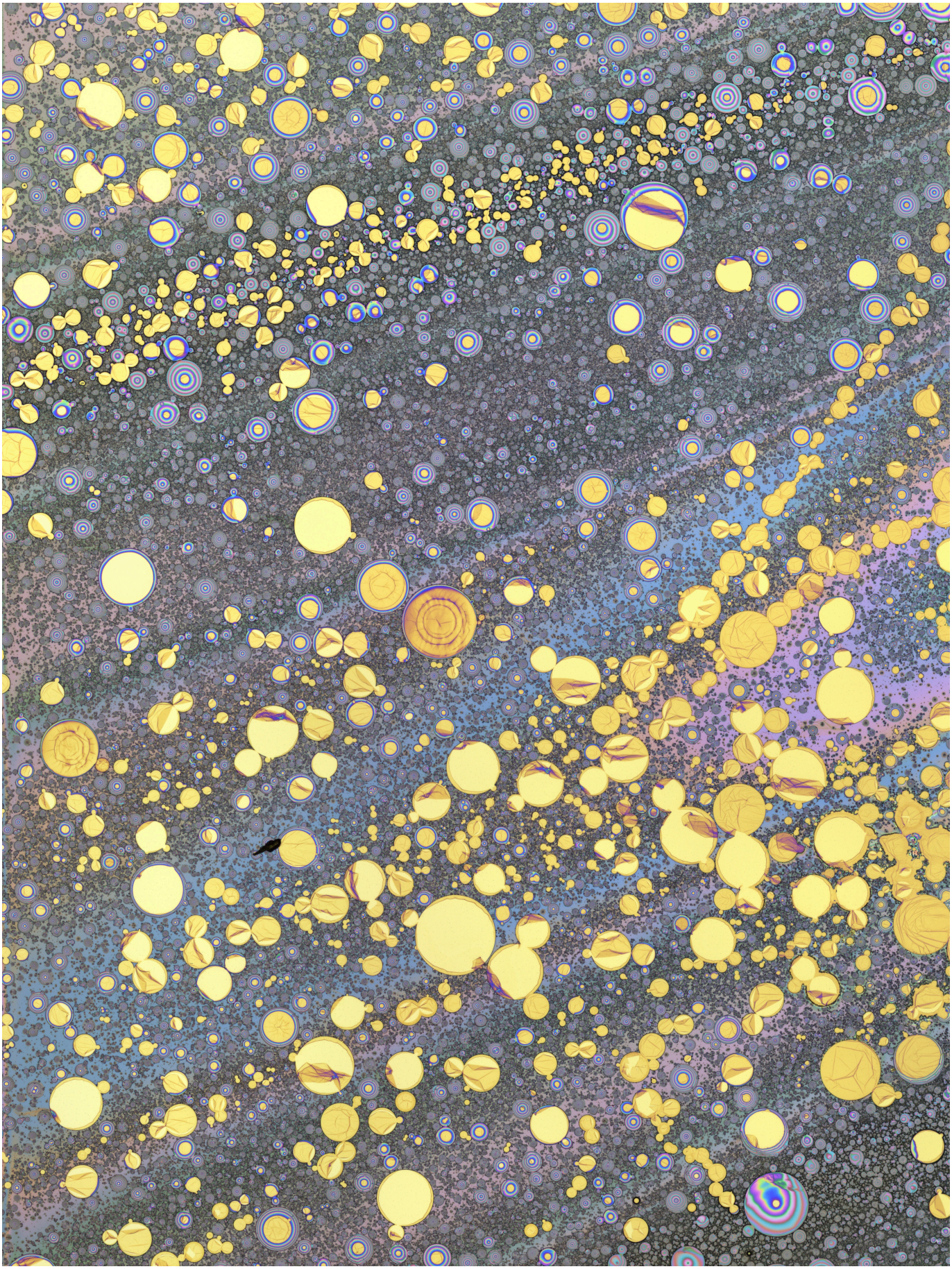


What a mess

300µm

Another glass-coated photoresist landscape, but this time the observed pattern is a result of a solvent immersion process, similar to Polymers 04. The glass layer had a high intrinsic stress, which resulted in the layer shattering when the polymer supporting it was removed. Here we probably see different amounts of photoresist still trapped under the glassy layer, resulting in a range of colours. It is fun to think about that this colour is almost exclusively structural in origin.

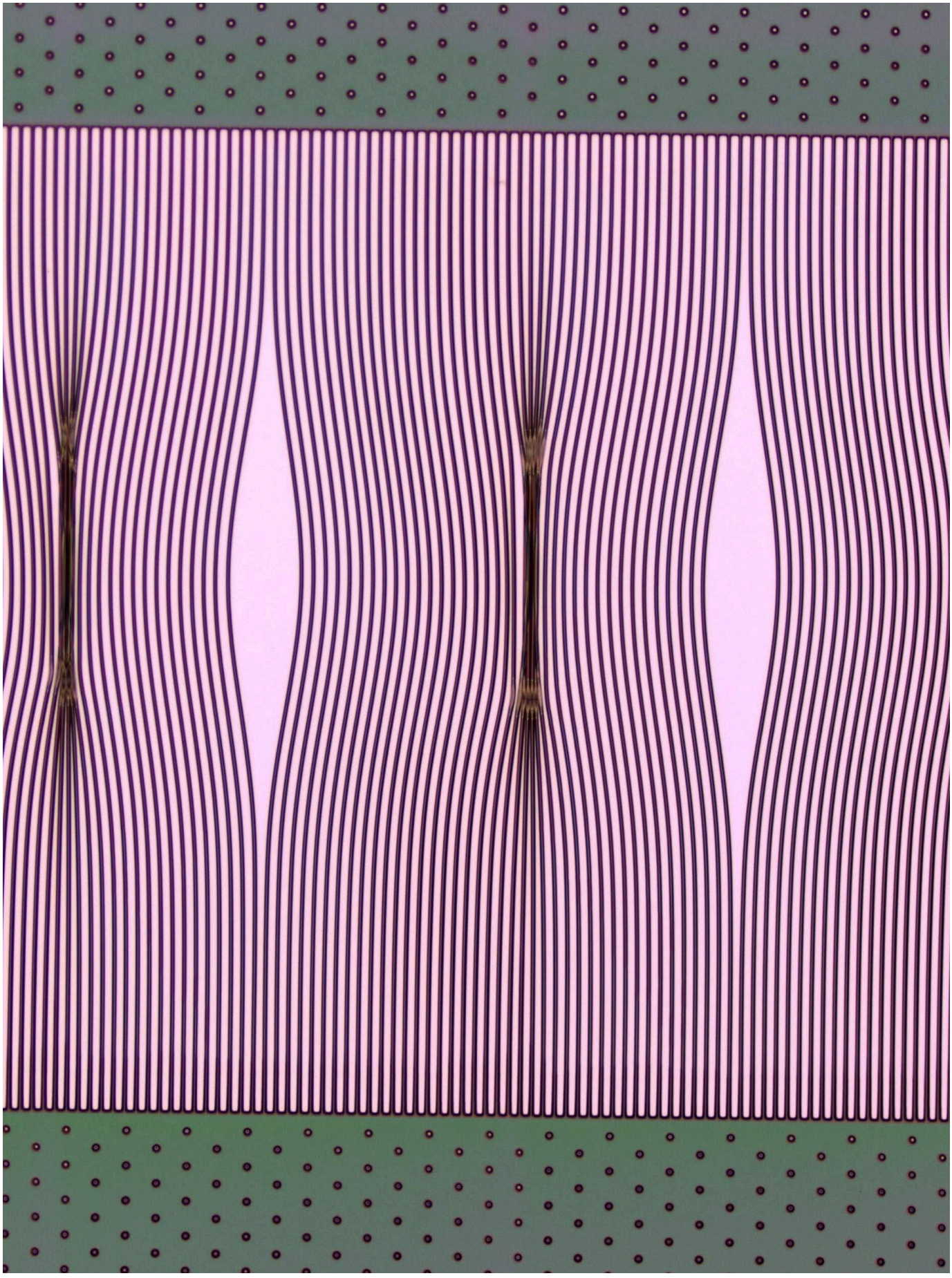
Olympus BX51



Starry Night

300µm

In this cleanroom take on an iconic painting, photoresist was exposed to a plasma etching process with CHF_3, which will fluorinate the top layer of the resist. This 'crust' then becomes insoluble in typical solvents. In an unusual workflow, the resist layer was put back into its developer solution *after* the plasma process. The photoresist, now locally fluorinated and exposed to the light and heat of the plasma process, produced this nighttime sky full of radiant photoresist blisters.

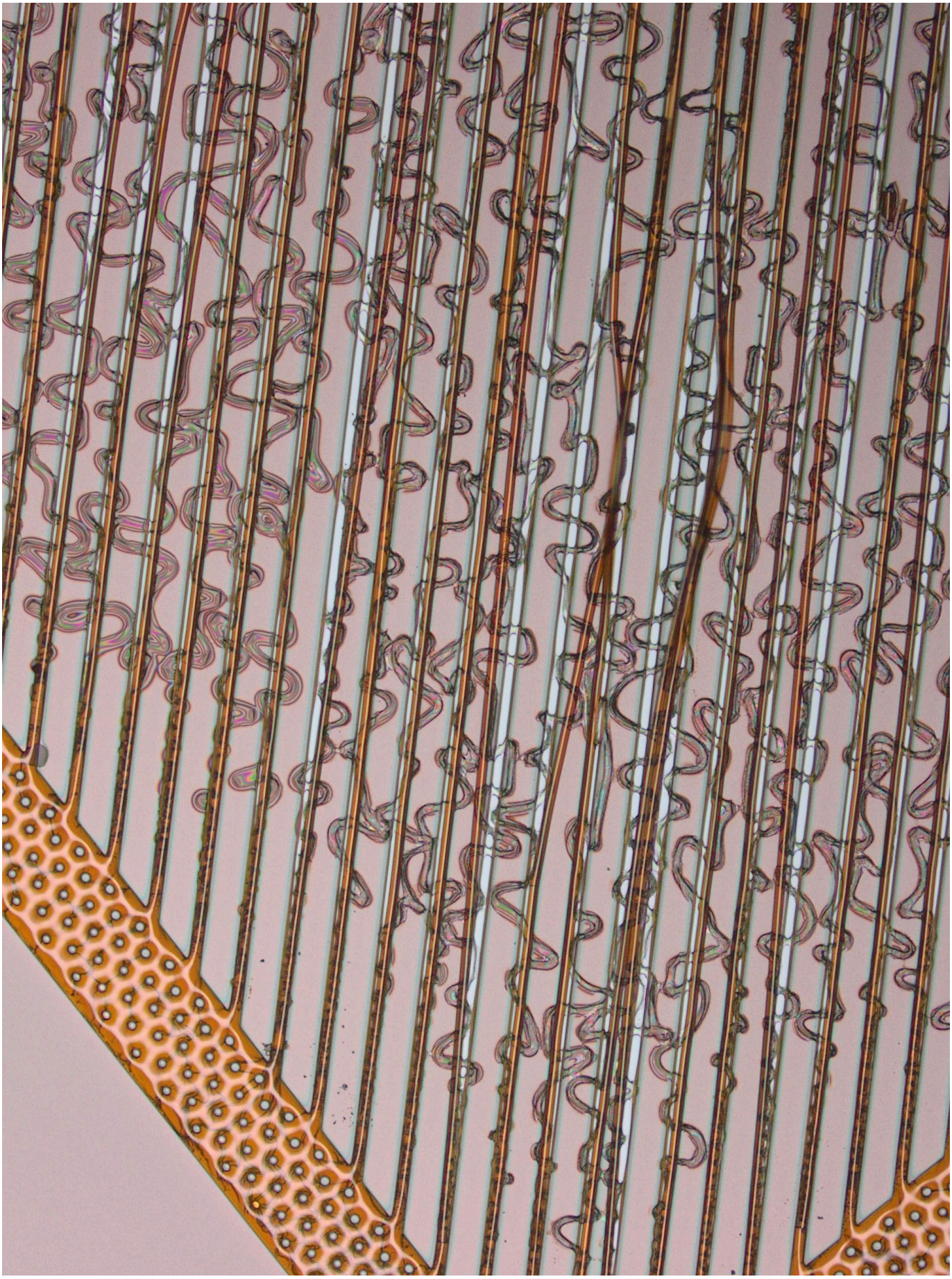


The Parting of the Sea

300µm

Whether your layer is organic or inorganic, it is important to make sure that the deposited layer is able to adhere well to the layer before it. If you fail to do so for resist, as in this image, you may find that parts of your pattern delaminate from the surface.In this case, the long slender resist lines (green/brown) have completely detached from the surface, and are held down only at their ends. Liquid flows during development have resulted in the lines moving together as groups from their original (perfectly vertical) orientation.

Olympus BX51

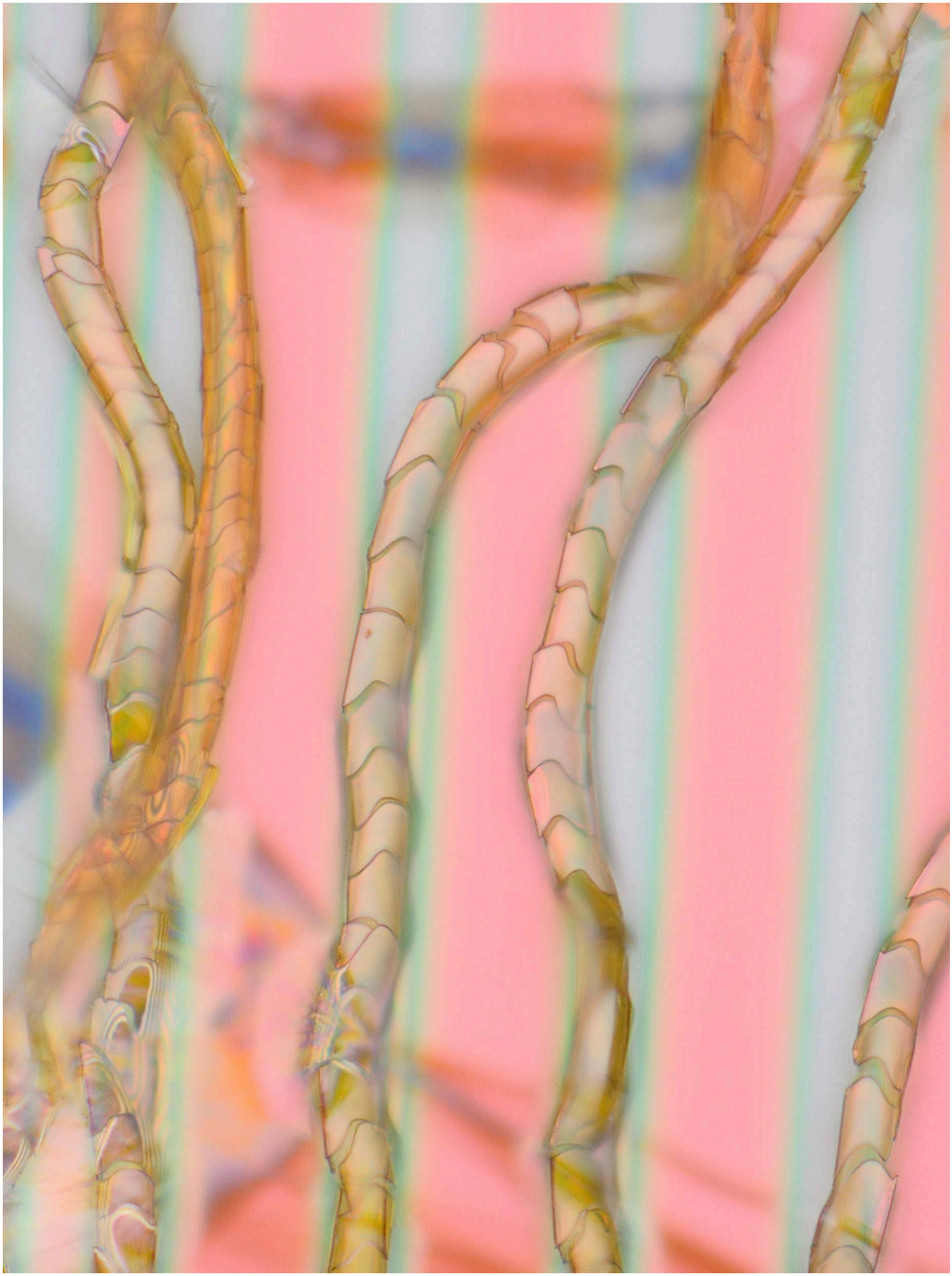


Umbilical Cords

50µm

This landscape was another unexpected one. Photoresist lines were encapsulated in a silicon carbide layer, which was a little too hot for the resist. The gas formation must have ended up forming cracks in the silicon carbide — as after immersion in organic solvent these long photoresist umbilical Cords were seen. I suspect the photoresist somehow ended up swelling but not dissolving in the organic solvent, as the contour length of the lines is much longer than the length of the channel itself.

Olympus BX51



Micro Annelids

50µm

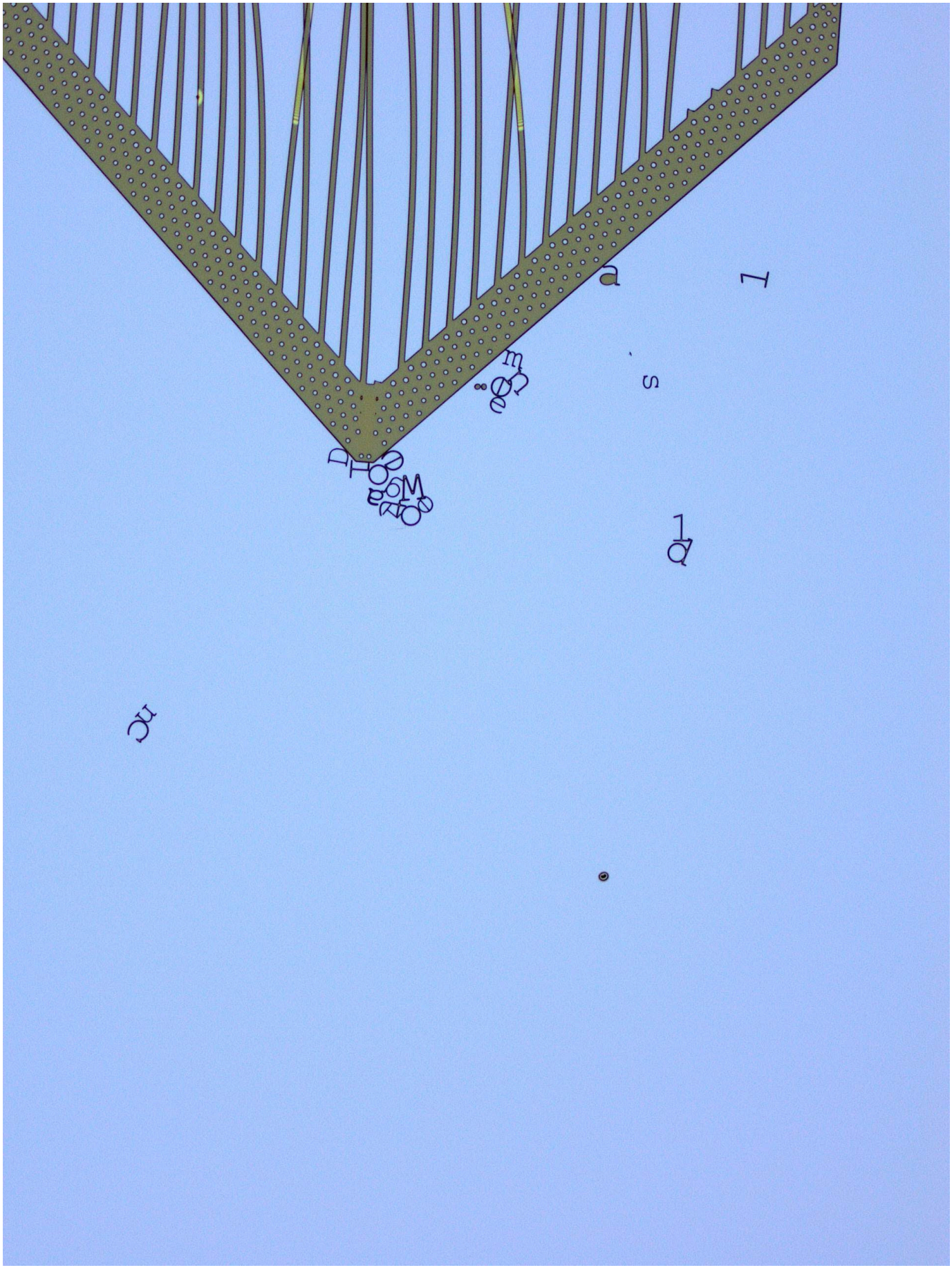
While the stackup in this image is identical to the image on the left, in this case, the brittle silicon carbide layer ended up cracking along the channel length, with the (partially) insoluble polymer remaining inside. This polymer backbone kept the structures together; forming a brand new kind of segmented worm.



Houston, we have a leak

100µm

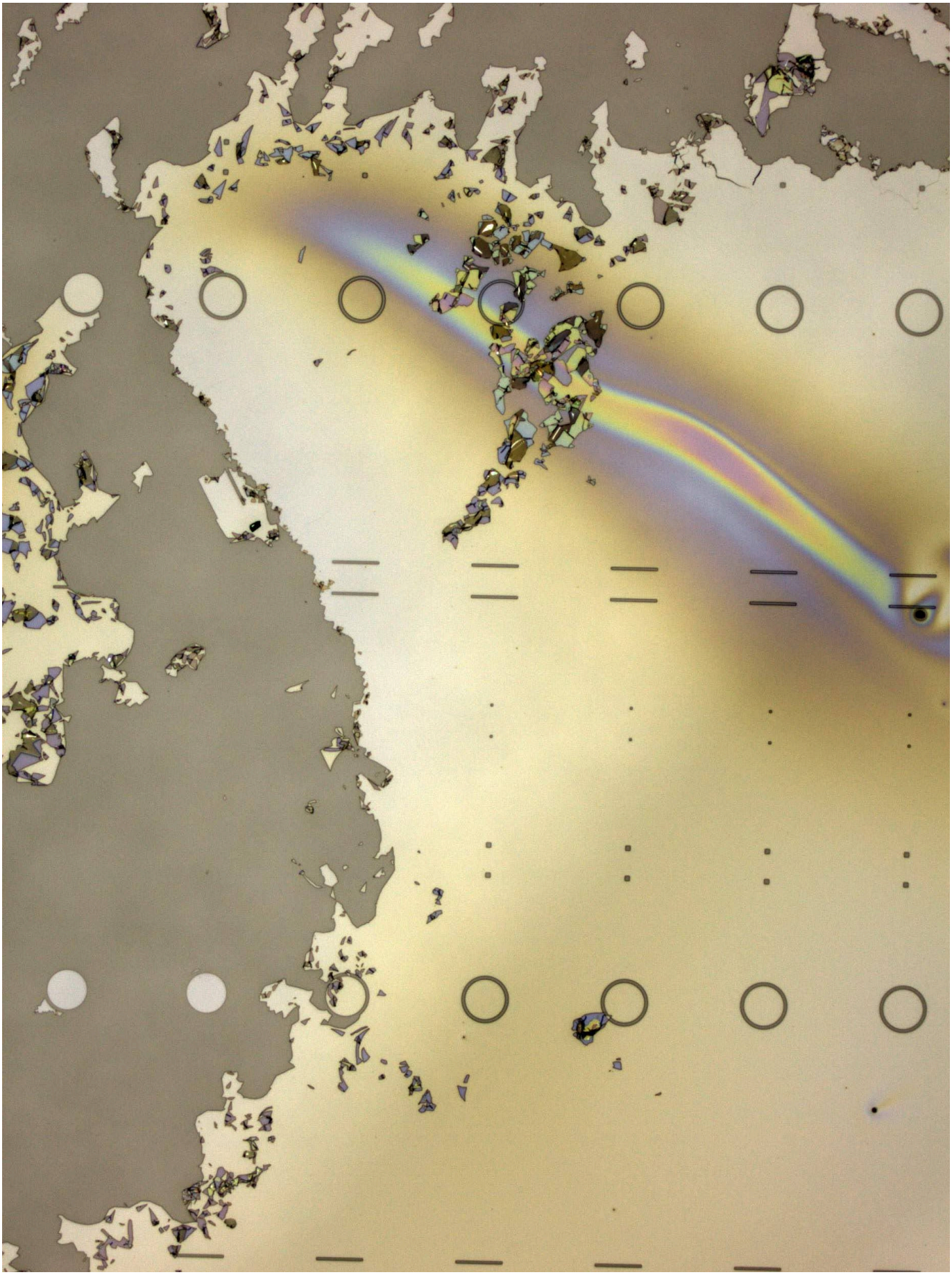
Another example of adhesion issues of silicon carbide on aluminium oxide. Here the dark rectangular opening was etched through from the back of the wafer, allowing water to enter the channels. The adhesion of the carbide was extremely poor, causing the water to start creeping in between the silicon carbide and aluminium oxide layer. The adhesion problems were likely exacerbated by a long O_2 plasma process. Needless to say, these devices did not make for good microfluidic devices.



The Photoresist Oracle

200µm

What does the future hold for your microfabrication efforts? Wise men would advise you to consult the photoresist oracle. Piece together the letters dispersed on the wafer surface, and learn of your fate. The fate that the resist oracle read to me was short and simple: “If you don’t improve your photoresist adhesion, you will get many more fortune tellings in your future.”

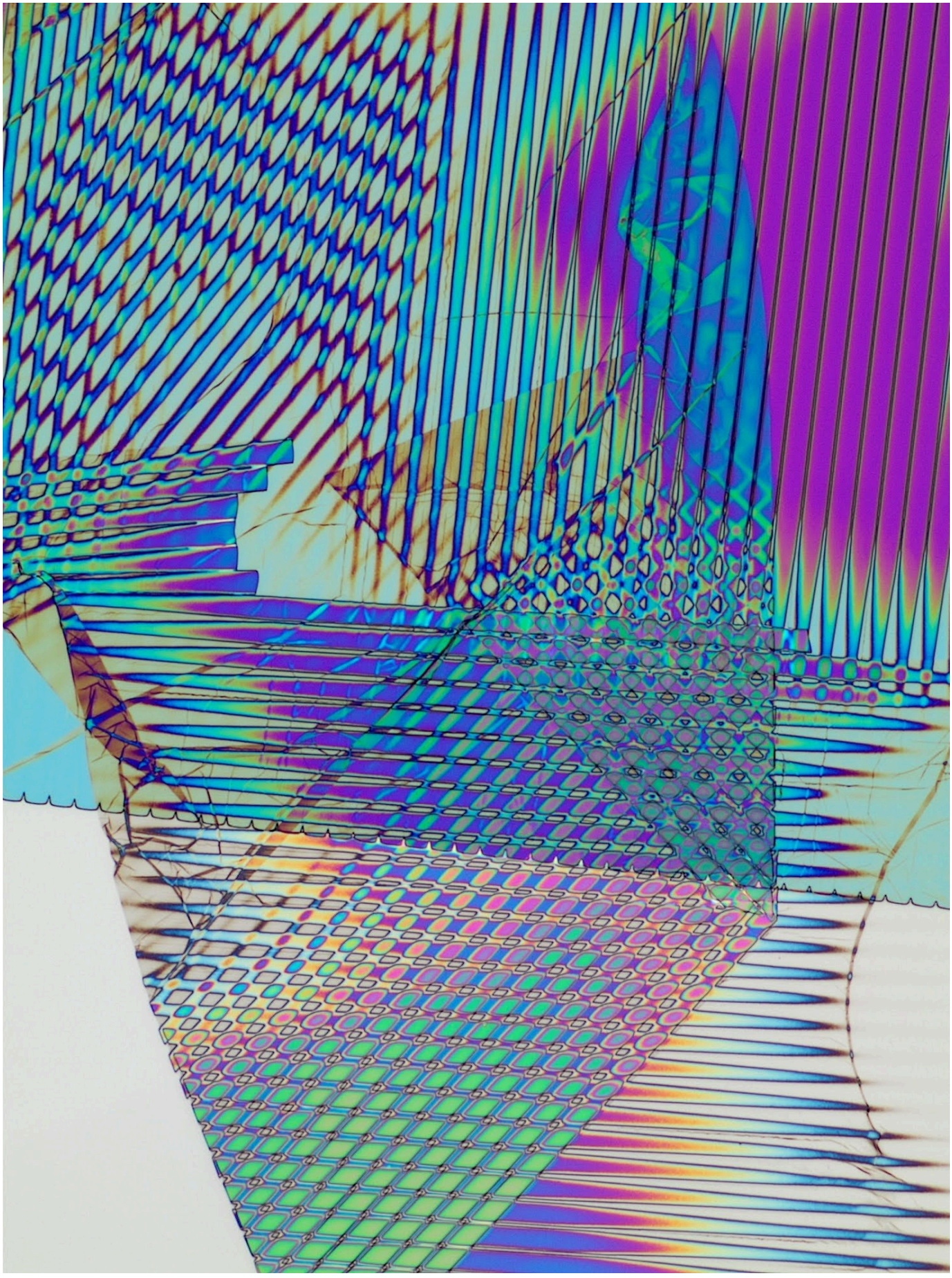


Letting Go

100µm

In cases where adhesion to a substrate is poor, small sections of photoresist will certainly delaminate from the surface, while larger areas tend to be able to stay attached. Sometimes adhesion is extremely poor and even large sections of resist decide to leave the premises. Here an ultrathin (less than 200nm) layer of photoresist decided it did not like sticking to a piece of coverglass—fragmenting itself during development.

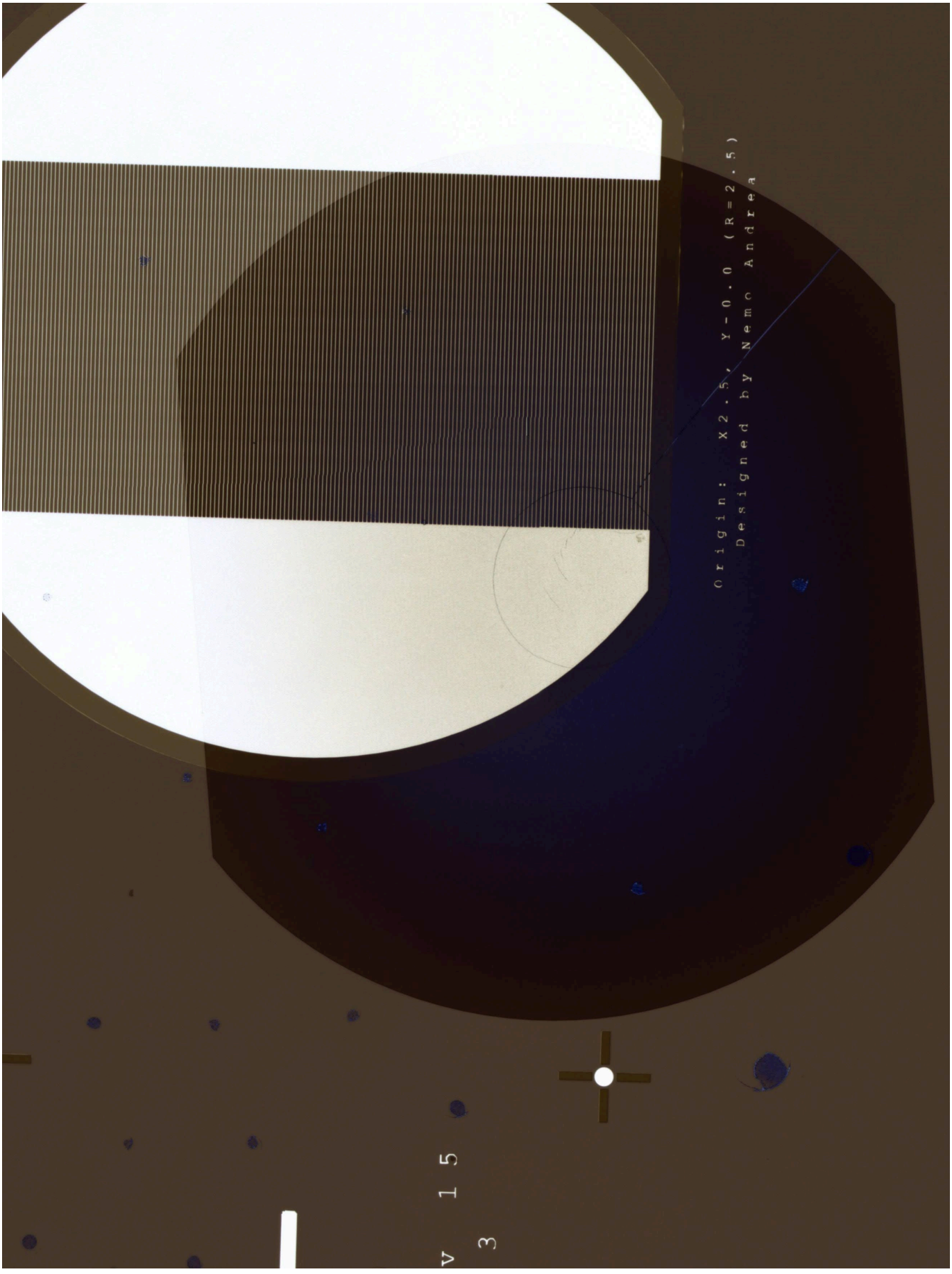
Olympus BX51



Crisscross

100µm

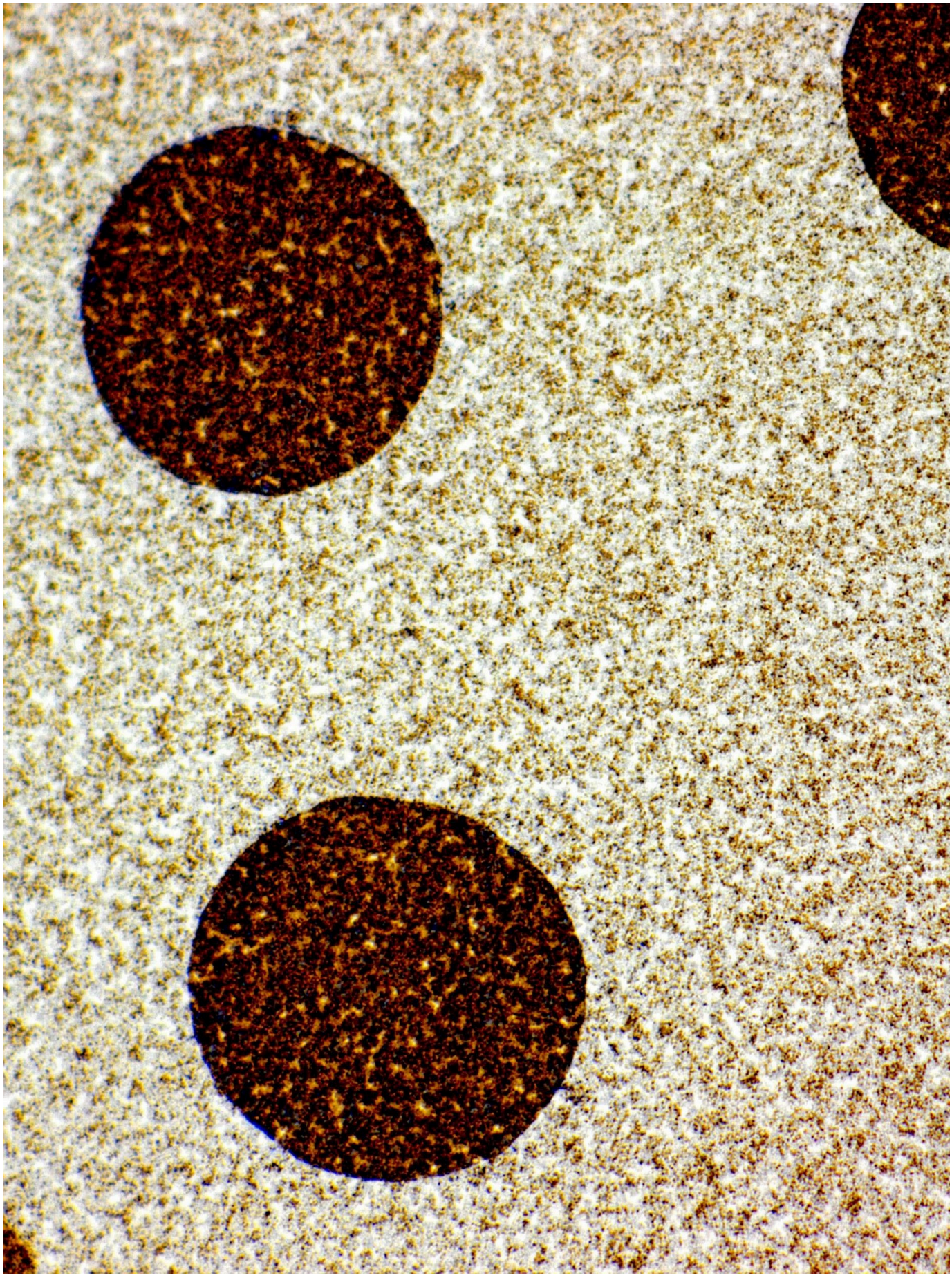
A case where photoresist decided to let go from its original location, but felt homesick and returned; stacking onto itself in a crisscross pattern. Notice How the effect is quite similar to Inorganic 06. Photoresist may produce different colours compared to inorganic materials due to their intrinsic absorption towards the blue end of the visible light spectrum.



Not All Those Who Wander Are Lost

100µm

In our attempts to grow graphene directly on the silicon substrate, we used a molybdenum thin film catalyst. The molybdenum (with graphene on top) is then etched away, allowing the graphene to 'land' onto the device. While distance for the graphene to 'land' is *at most* a hundred nanometer, the whole film can still be displaced a large distance as we can see in the image. Just what we needed; wandering graphene layers.

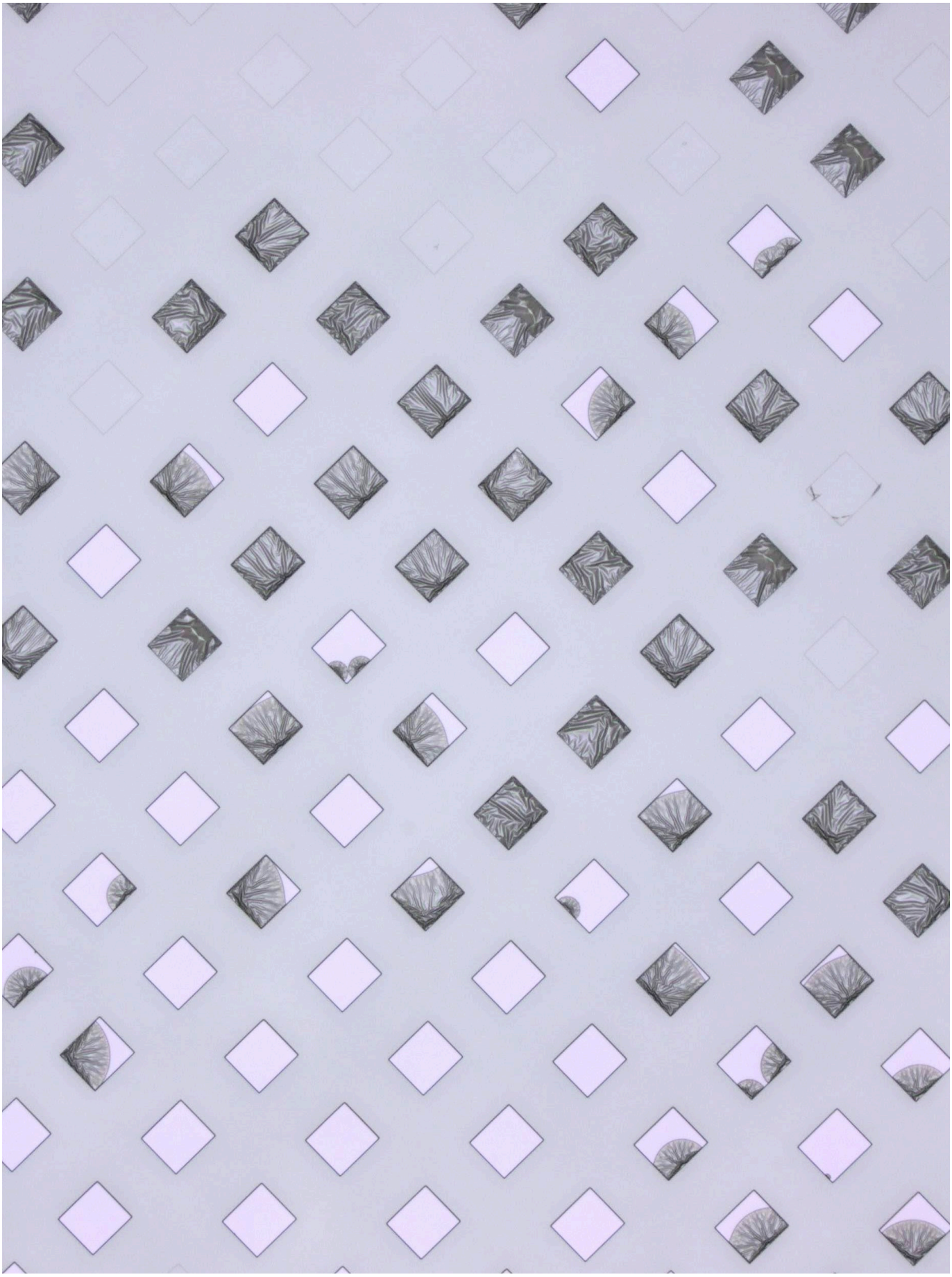


One Micro Pepperoni Pizza, Please

100µm

This delicious looking microscape was not produced in the world's smallest artizanal pizzeria, but instead shows a wafer surface after graphene CVD. Molybdenum was again used as the catalyst, and had been etched away in the image shown. The graphene/graphitic layer that was left after growth does not look promising. The 'Pepperoni slices' are either aluminium oxide blisters (formed due to the CVD heating), or points where the molybdenum alloyed with the silicon substrate.

Olympus BX51

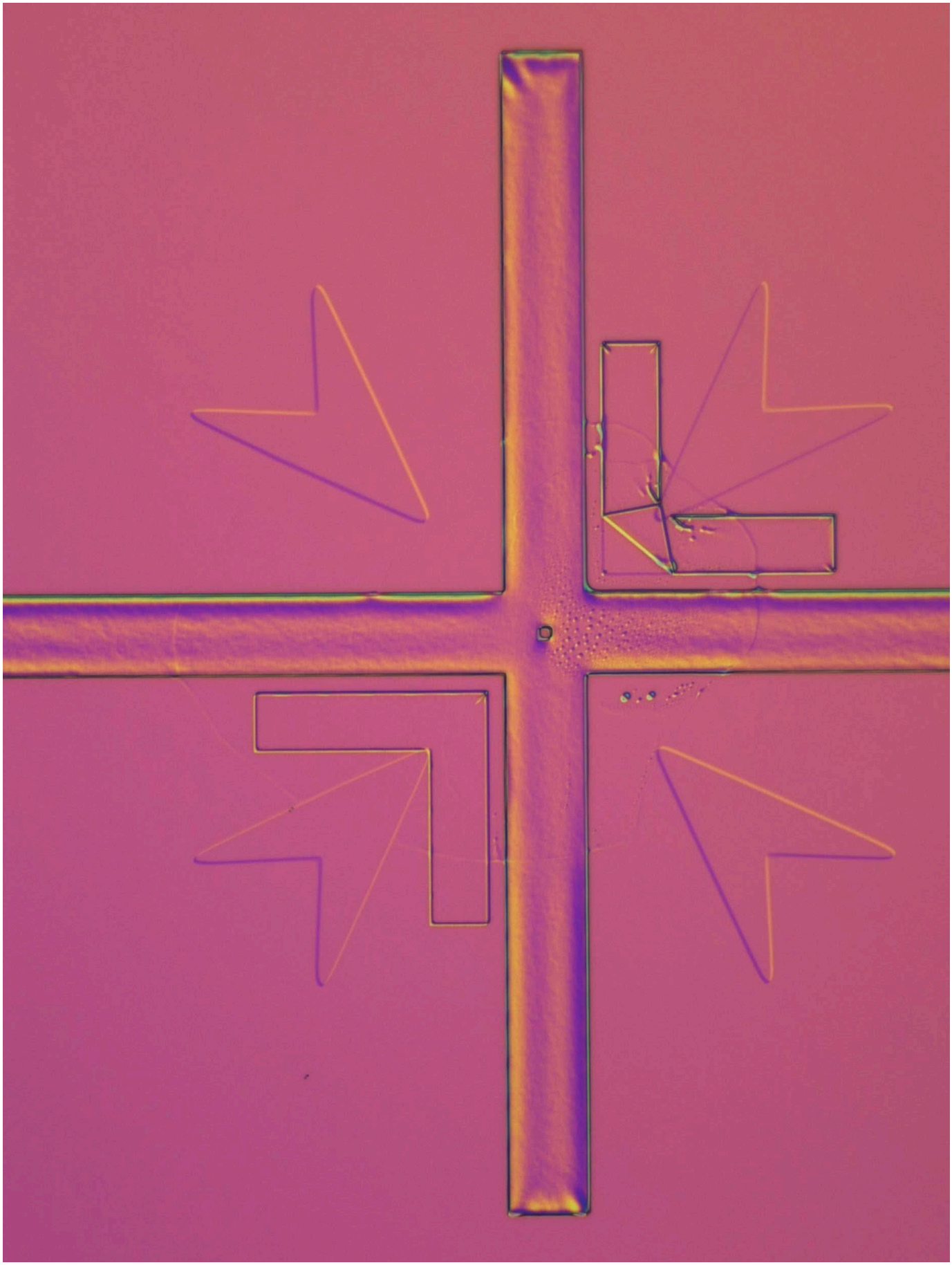


Manhattan

150µm

This image shows the same kind of ion-milled substrate as Inorganic 27, but with the photoresist mask still present. Here the photoresist was partially removed by ultrasonication in acetone; but the resist was determined to cling onto the substrate. The heating that is intrinsic to kinetic etching processes such as ion milling hardens the resist, making removal a challenge. We can see squares of succesful removal (same colour as background), areas of intact photoresist (white) and areas of wrinkled, partially delaminated resist.

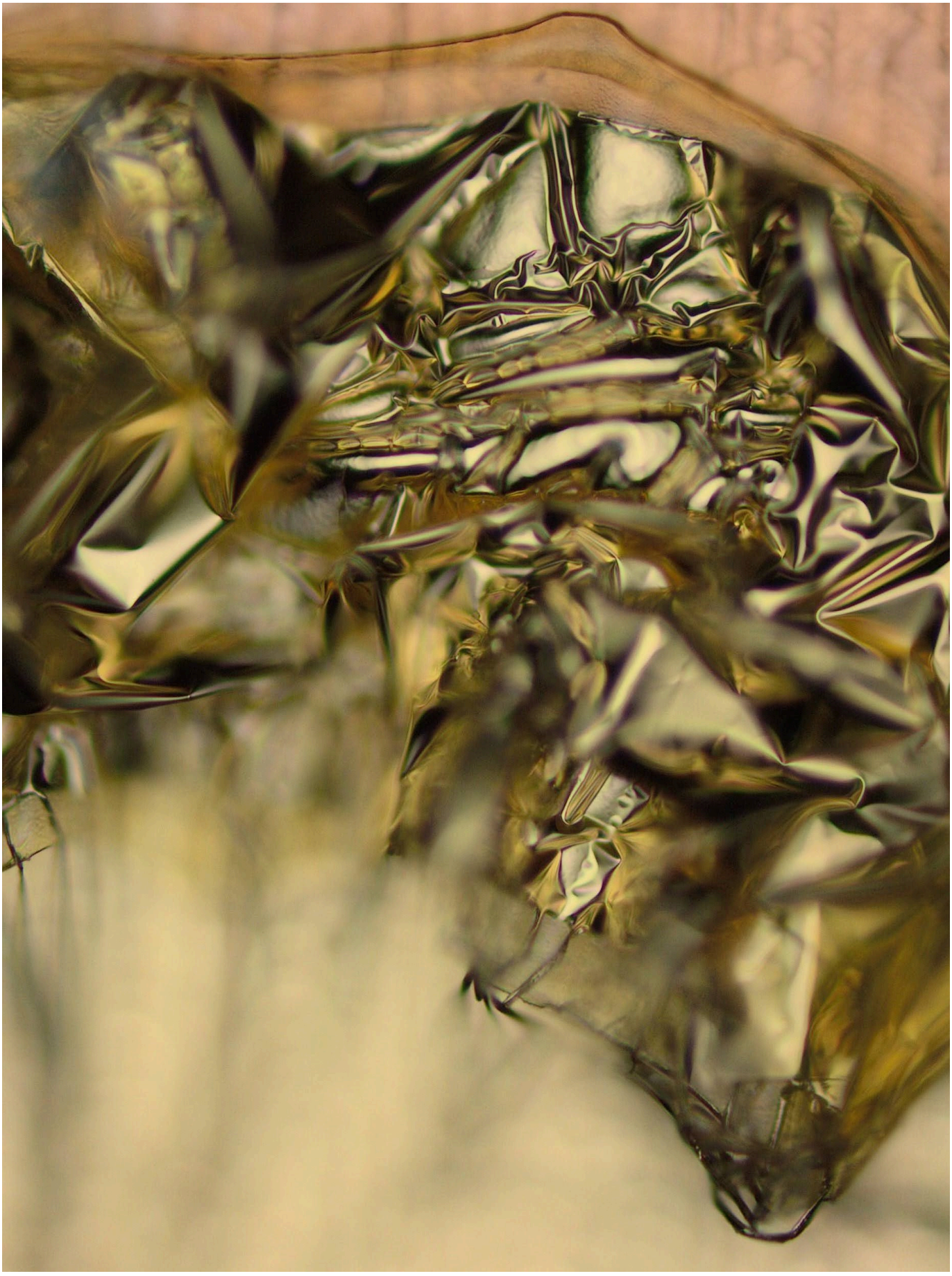
Olympus BX51



X Marks the Spot

150µm

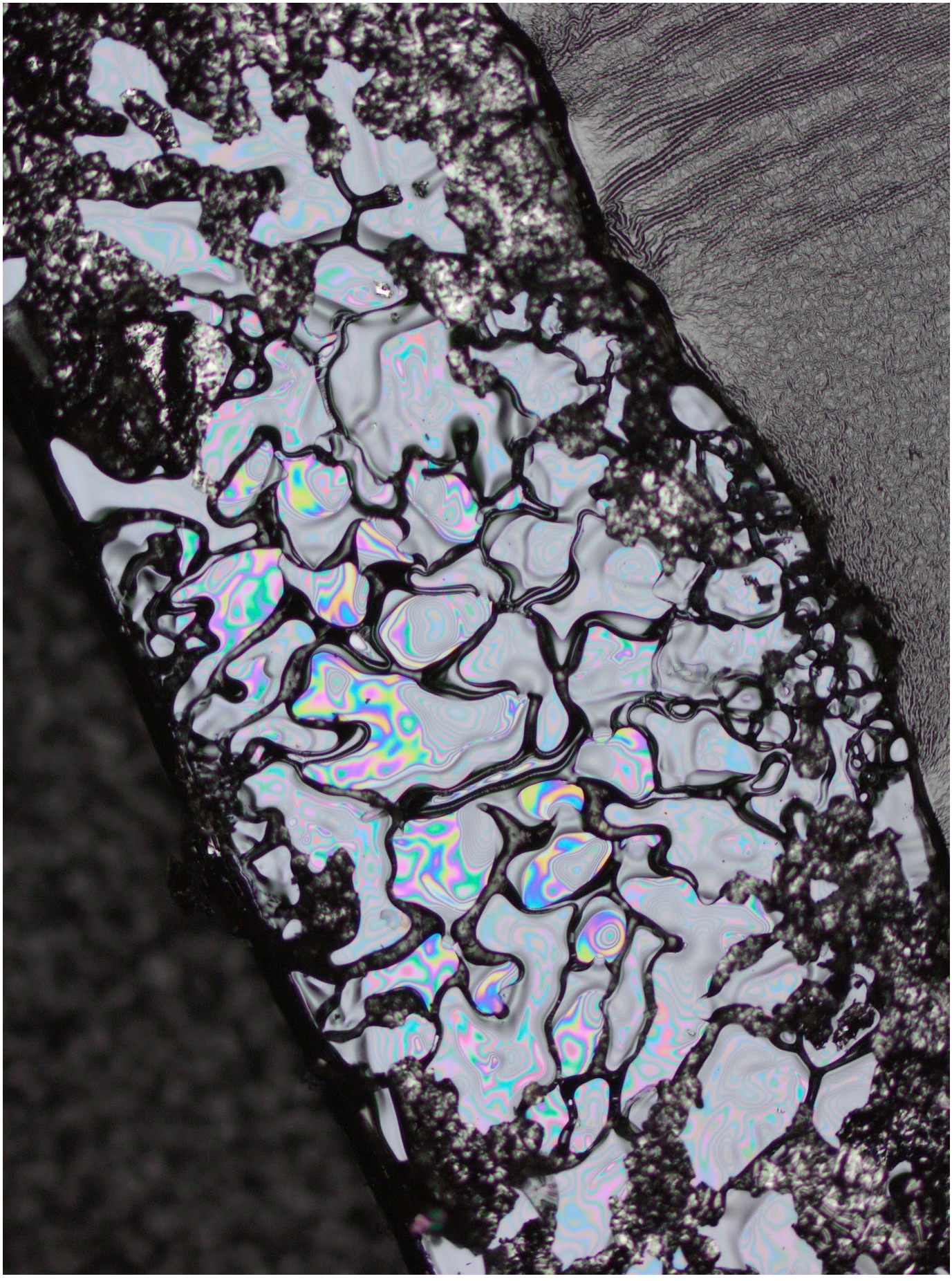
In order to accurately align the different layers of the device, marker features are introduced into the layer design, allowing the different exposures of the design to be accurately aligned. This differential interference contrast microscape shows one such marker structure, and the relative alignment of different layers. The arrows are features defined in molybdenum, while the slightly wrinkled features are photoresist features that have been partially dissolved by organic solvents.



All That Glitters

300µm

In order to pattern gold on graphene, we tried a lift-off approach, where the gold is deposited onto photoresist and then the resist is dissolved. Little did we realise that this negative resist will not dissolve, even in very strong organic solvents. All we really ended up doing is making a sandwich of photoresist and gold delaminate from the copper surface—at which point the intrinsic stress of the evaporated gold layer had free reign to crumple itself up.



Under Pressure

300µm

In order to ensure good sealing of the silicon wafer in the deep reactive ion etcher, the wafer is clamped down at the edges of the wafer. If this ends up clamping directly onto photoresist in the hot plasma chamber, the wafer will end up ‘glued’ to the plate. This will then result in either the wafer breaking during unloading, or an angry tool owner. I hoped a simple solution might be to sputter some (non-stick) molybdenum onto the photoresist. The clamping plate tore right through the thin molybdenum film. Back to the drawing board.

Olympus BX51

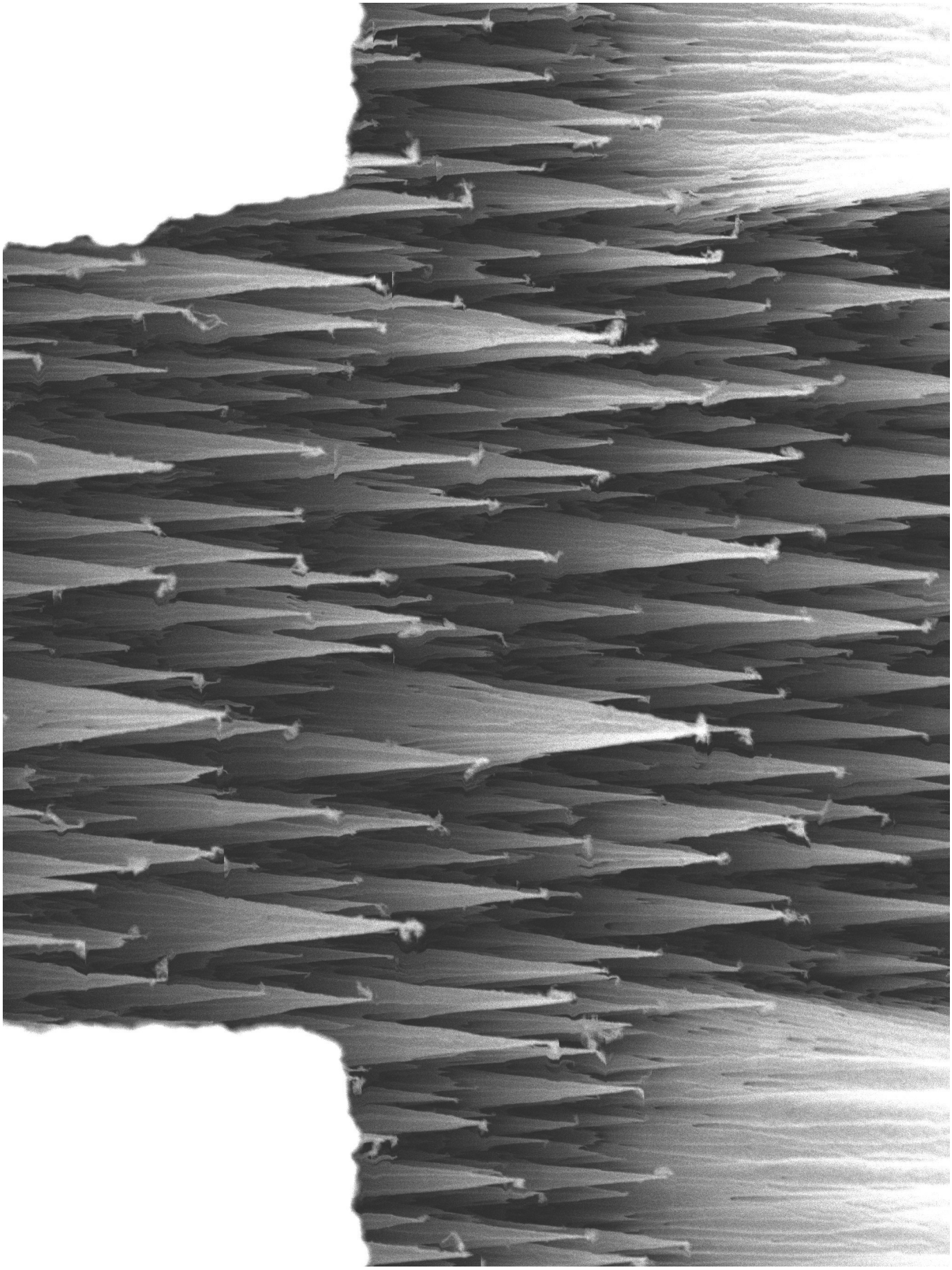
3. Electron Microscopy

Light microscopes are fantastic tools for characterising larger defects, but come with one big limitation: there is a limit to their resolution. For a typical microscope employing blue light, that diffraction limit is around 200nm. Even with the most expensive lens design, you cannot resolve features smaller than that limit. If you need to look at features smaller than that, or need to be sure that there are no defects that may not be resolvable with a light microscope, you would best turn to electron microscopy.

Electron microscopes use electrons, rather than photons for the imaging of the sample. Electrons can readily be accelerated to energies where their wavelength is well below that of visible light, while remaining focusable by lenses. With a short wavelength, the diffraction limit can be pushed well beyond the single nanometer range. The tradeoff is that electron microscopes are expensive instruments, and that the sample must be imaged in a vacuum environment. Routine inspection of a device is therefore best conducted with light microscopes, while electron microscopes are best left to the most critical steps of a fabrication, or important quality control steps of single devices.

The most apparent difference between the pictures of this section and that of the preceding ones will be the lack of colour. In the light microscopy images, we saw a large range of colours caused by how the different wavelengths of visible light interact with, and are attenuated by, the sample. While there is a small range in wavelength spread in the electron beam, there is not much useful information in the difference in how these different energies propagate through the sample. To make matters worse, focussing different electron energies in the same spot is a tricky affair, and hence wavelength spread in the illumination source is minimised as a matter of microscope design. Electron microscopy is therefore monochromatic: only a difference in the count of electrons that hit the detector makes up the observed contrast.

The contrast and general appearance of the image also depend on the kind of electron microscope used. The most ‘picture-like’ electron microscopes are scanning electron microscopes (SEMs); which produce an image by scanning a focused electron beam across the sample, and produce images with lighter and darker patches that appear almost like shadows in a typical photograph. For applications that aim to elucidate the smallest features down to single atoms, a transmission electon microscope (TEM) can be used. In these images, a flat projection of the sample is produced, which is usually of lesser aesthetic interest and certainly of lower familiarity.

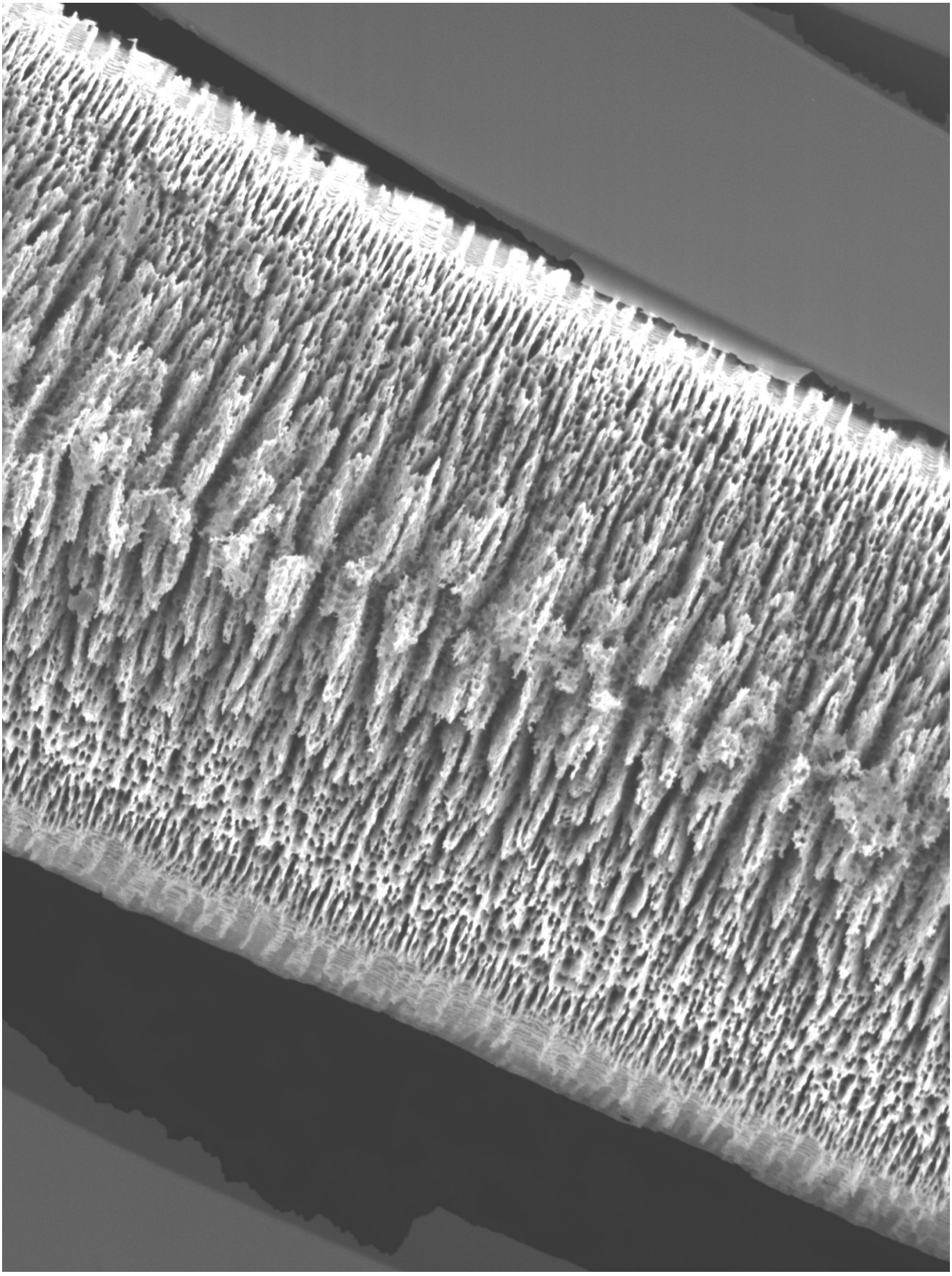


Once Upon a Time...

15µm

Once upon a time, in a microscale kingdom far far away, a thousand little castle roofs housed a thousand little royal courts. These silicon spikes were formed by a poorly tuned deep reactive ion etch process. In this case, there was both a little too much deposition of the C_4F_8 polymer, and the possibly the presence of micromasking elements. These little micromasked area then form slender silicon spikes. These structures function like tiny beam dumps; trapping light rays that enter the area; making the structures appear black when viewed by eye.

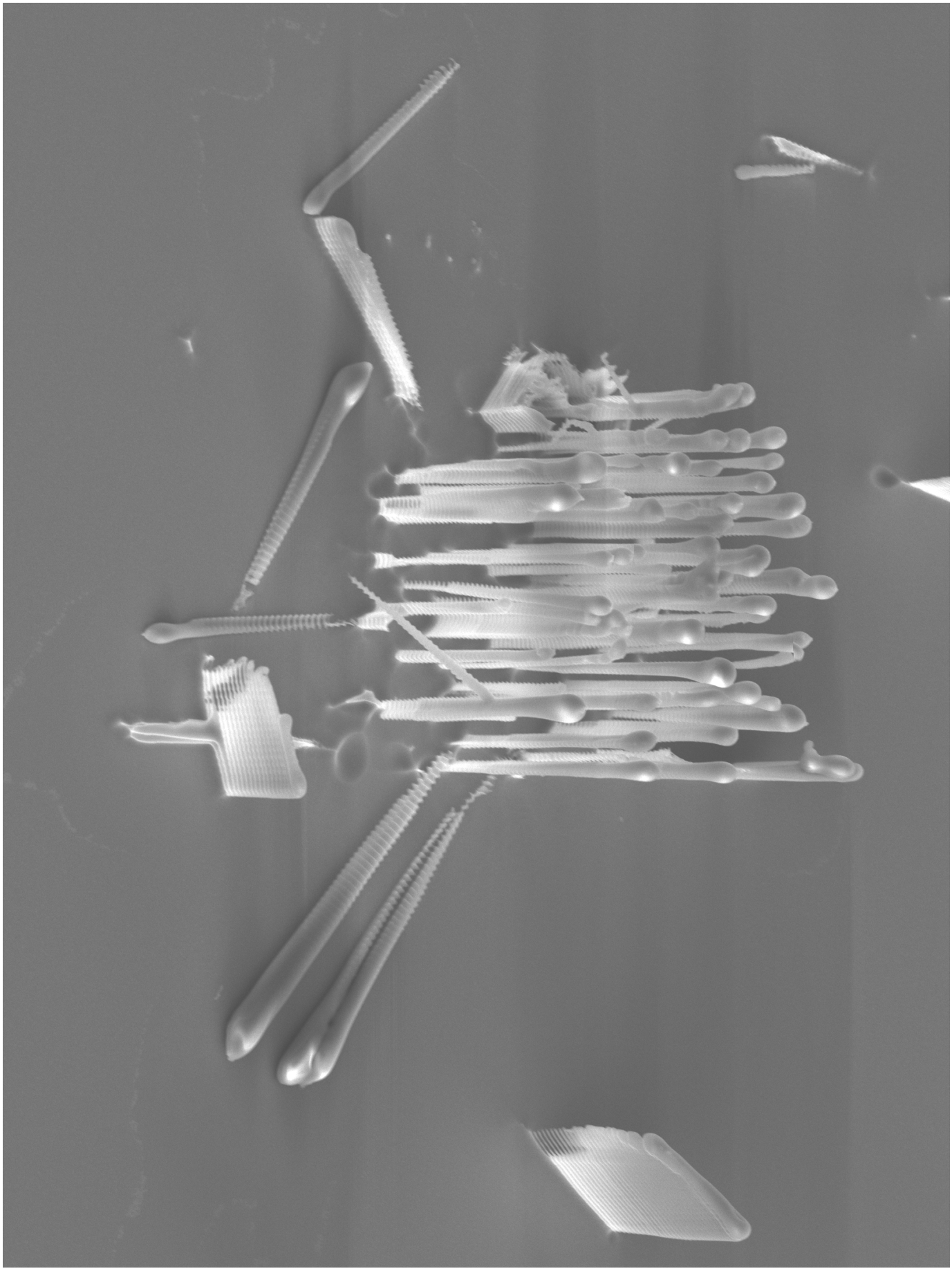
FEI Nova Nanosem 450



Porous Undercut

10µm

These porous silicon structures were formed during deep reactive ion etching, when electric charge built up on the stop layer. As the etching process involves the acceleration of charged particles towards the material to be etched, charge buildup will deflect the etching materials; resulting in sideways etching. In the image, we also see the layer responsible for the buildup: thin aluminium oxide membranes (dark gray).

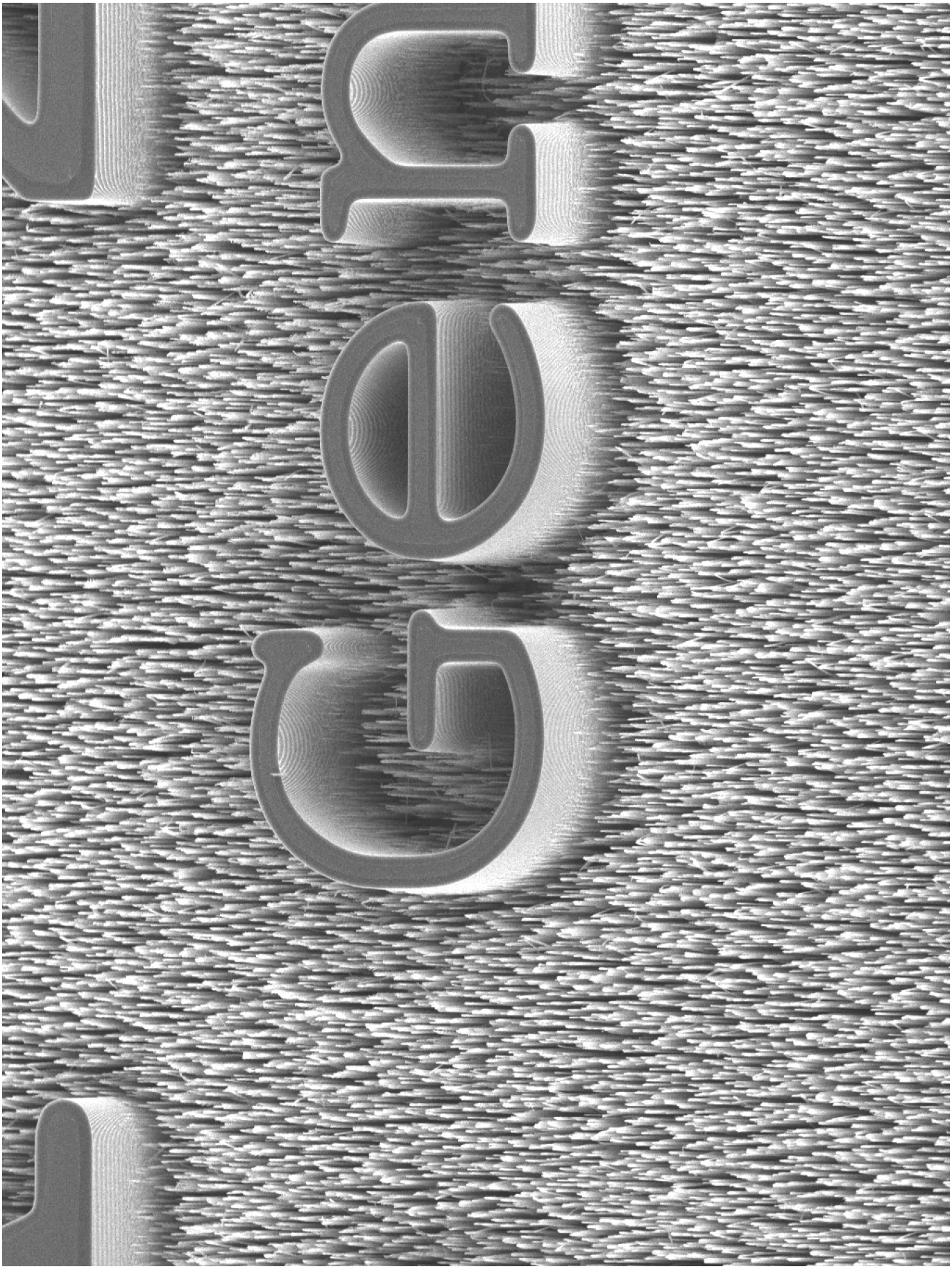


Springtime

10µm

The cycles of deposition and etching of deep reactive ion etching produce a distinct ‘scalloped’ (ribbed) texture on the sidewall of etched silicon. In this sample, which would become a PDMS microfluidic mold, this particualr remote area of the silicon wafer had some micromasking. Some unknown contaminant ended up preventing etching in a few spots, resulting in these thin stalks. It reminds me of plants sprouting in springtime.

FEI Nova Nanosem 450

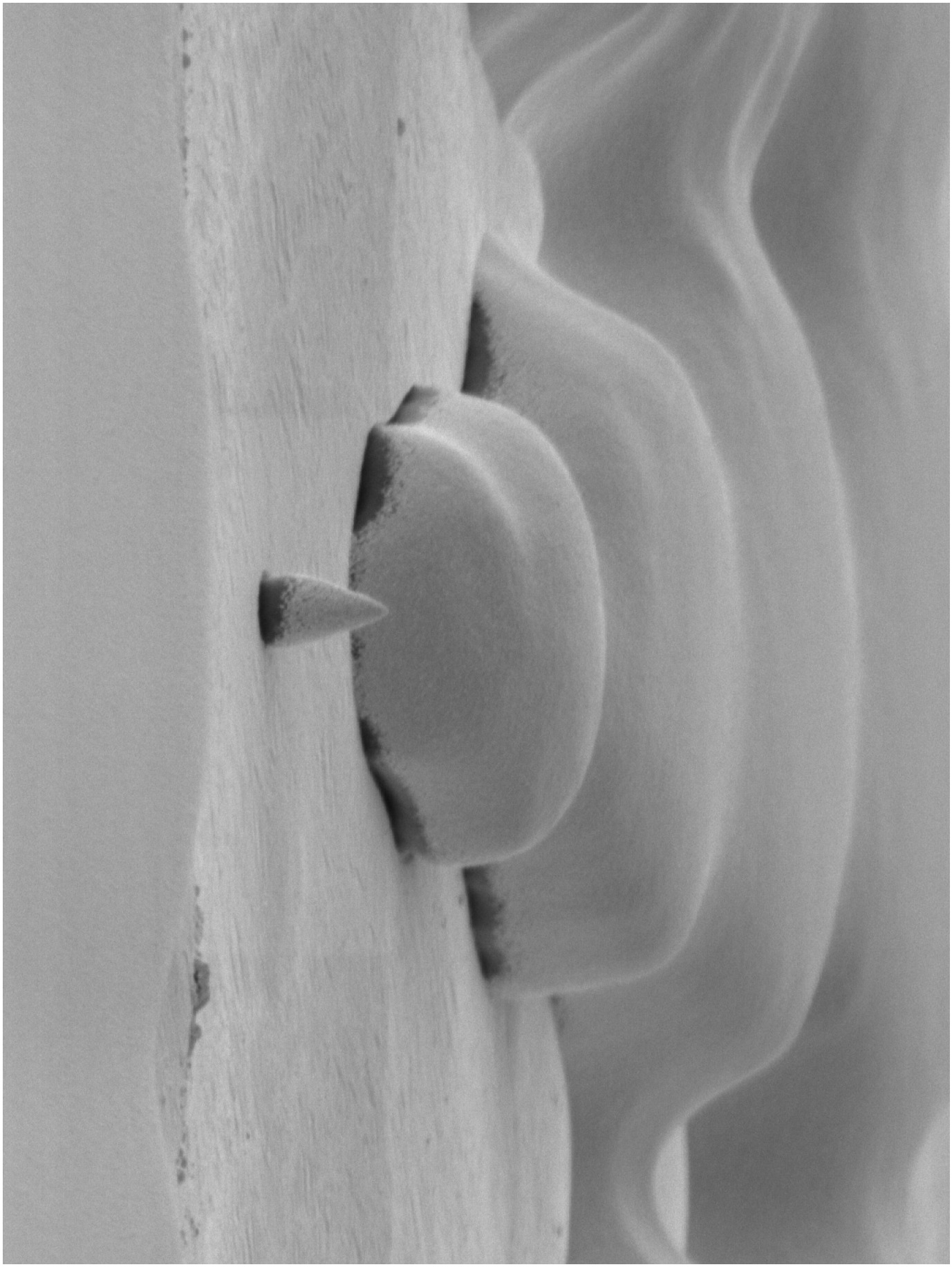


High Contrast Typography

50µm

In designing a PDMS template wafer, it is usually smart to add some metadata to the wafer design to allow traceability in the future. Here, the deep reactive ion etch process was unbalanced in the same way as Electron Microscopy 01, and the etched area was not smooth, but full of spikes. The scalloping on the sidewall of the letters is, somewhat surprisingly, entirely intact.

FEI Nova Nanosem 450

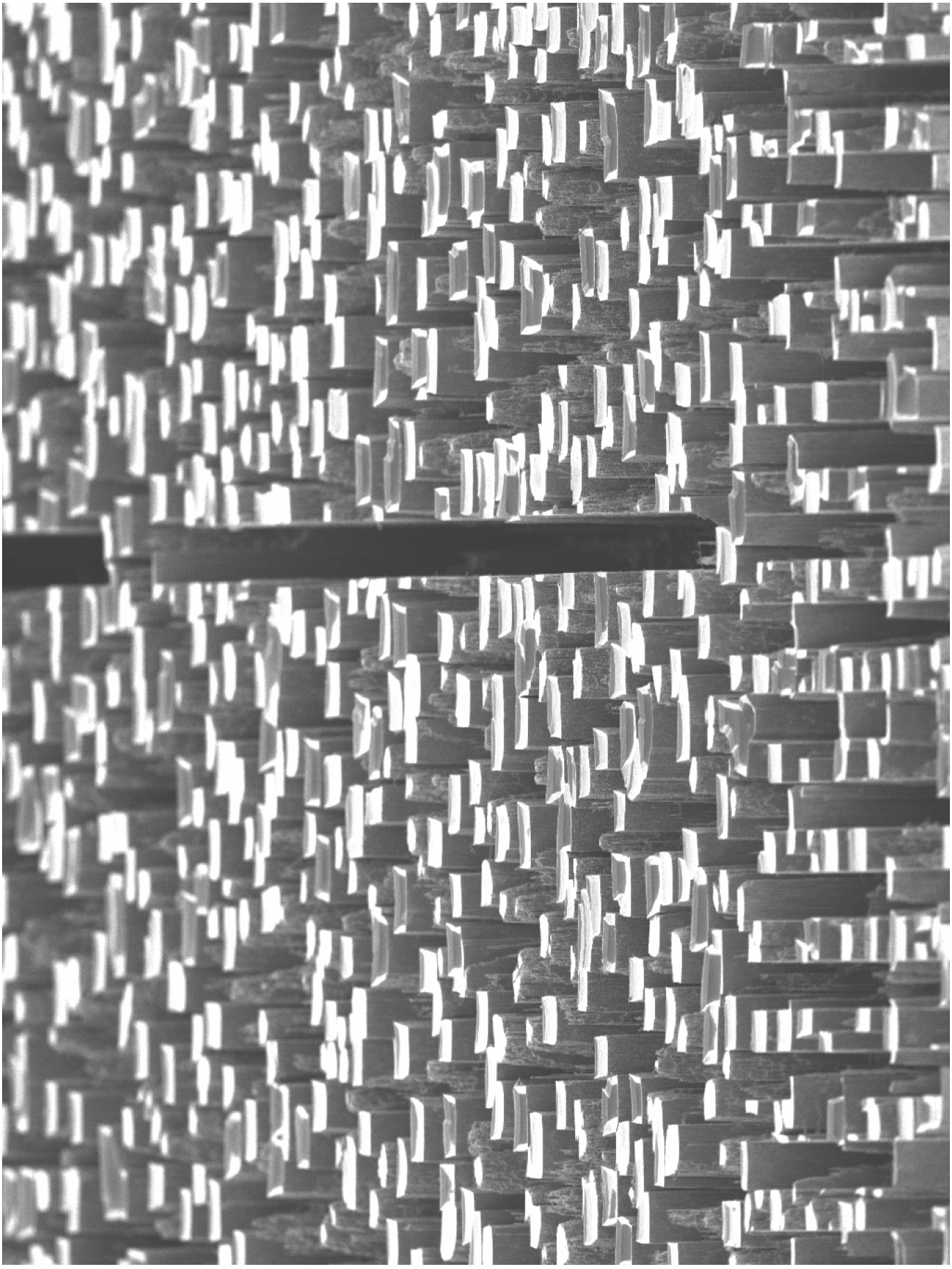


Christmas Season

2µm

One idea we had early on was to form gold-on-graphene structures. To realise this, we tried to take a lift-off approach, evaporating gold onto photoresist. In this example, underexposure and dark erosion of the (negative) photoresist resulted in the formation of structures smaller than the resolution of the lithography system. The ‘tree’ is around 500nm wide. This image is taken after gold evaporation; with the conductive gold ‘snow’ layer showing up as a lighter shade of gray than the non-conductive photoresist (dark gray).

FEI Nova Nanosem 450

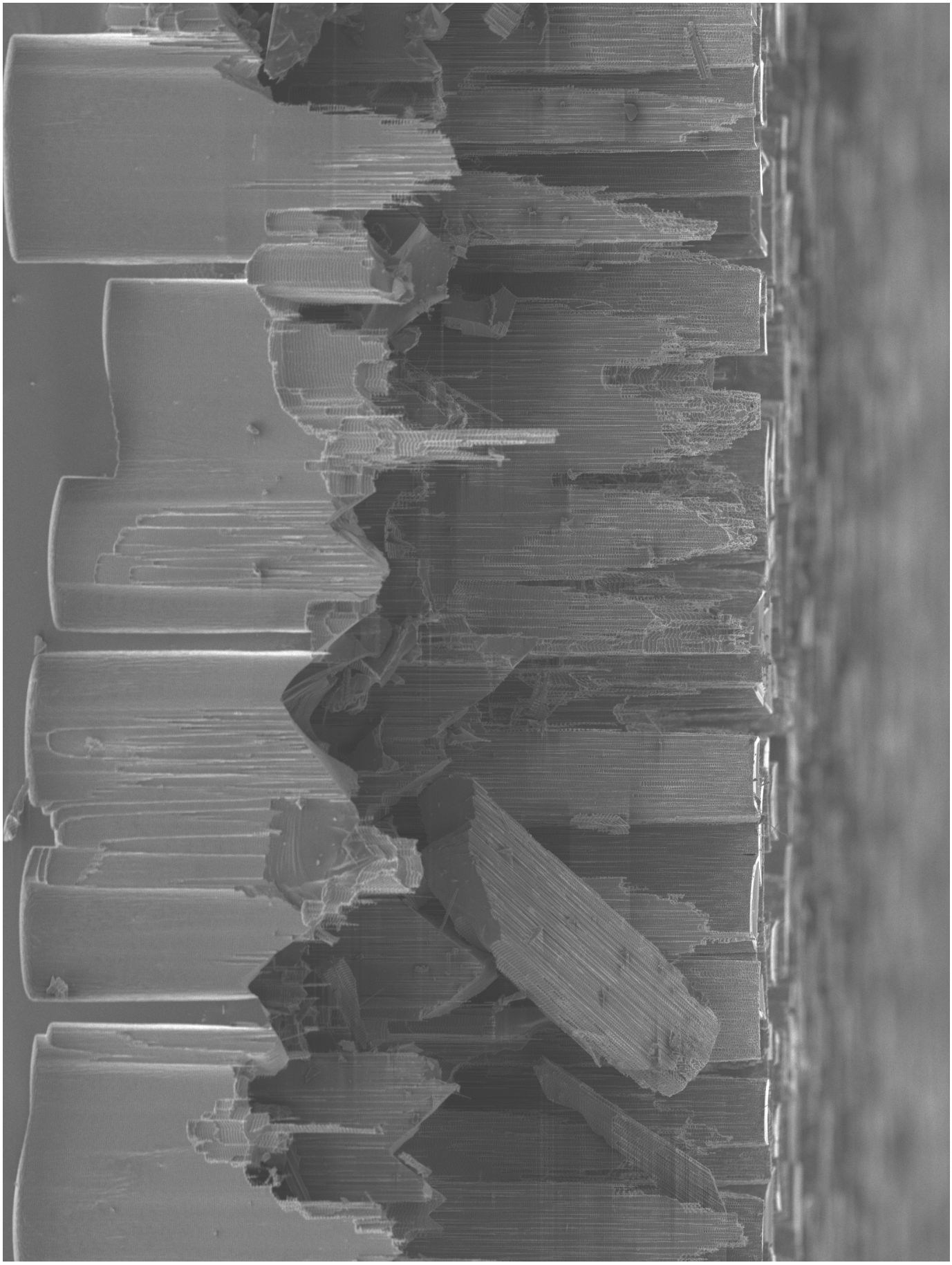


Central Park

50µm

Silicon wafers are cut from a large, synthetically grown, single crystal boule. After cutting, the cut surface must be mechanically polished down to the required roughness. Since a lot of devices are only built on one side of the wafer, wafers can be purchased with only one side polished; the other remaining rough. This is the unpolished side of a wafer, after a deep reactive ion etch process etched deep, elongated trenches into the wafer. One thing is certain: that is a lot of valuable micro real estate.

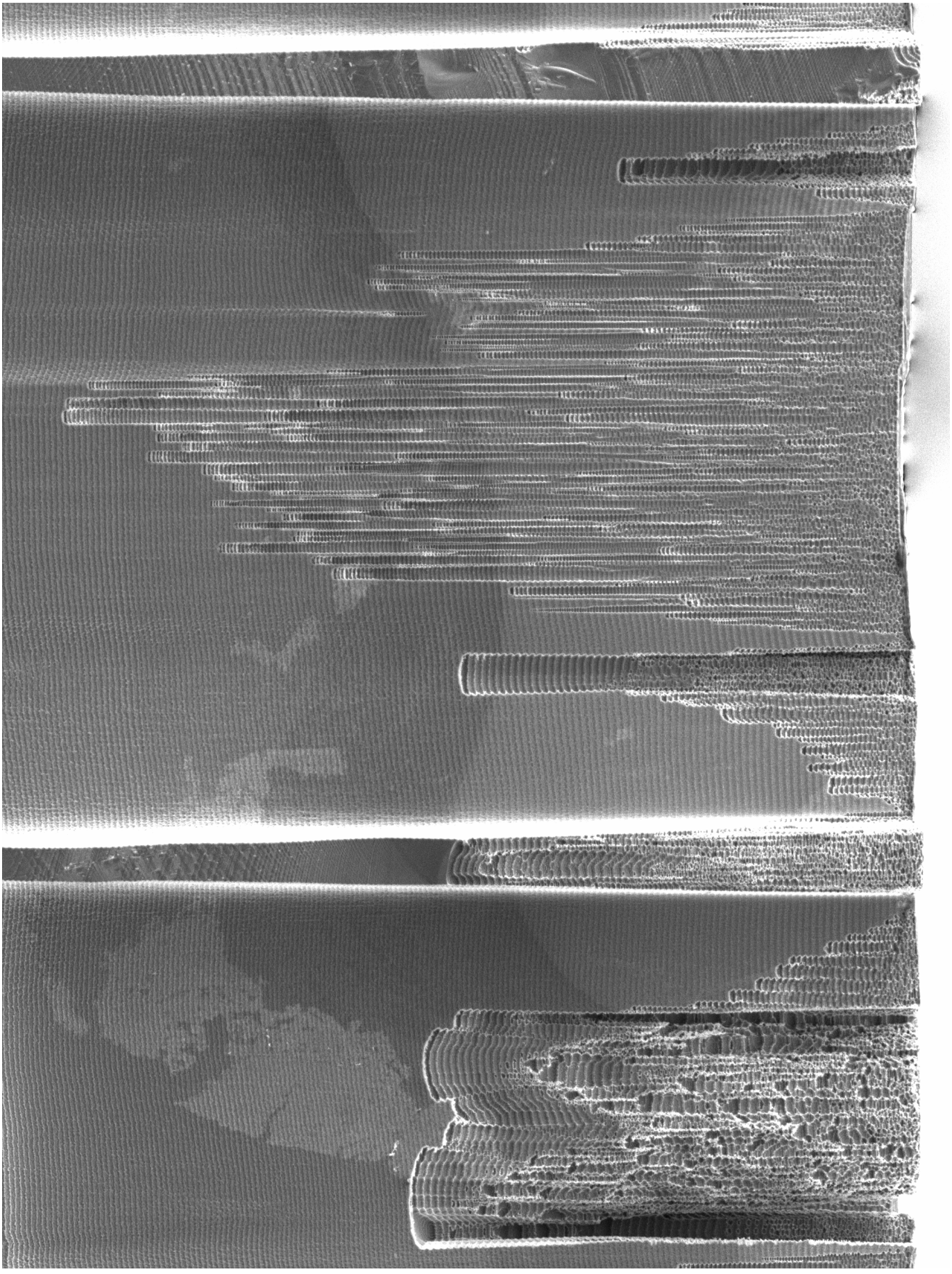
FEI Nova Nanosem 450



Armageddon

50µm

The same substrate as on the page on the left, but now looking at a cross-section of the etched trenches. The mechanical dicing of the silicon substrate has, in this area, ended up breaking many of the fragile thin silicon structures. It would be wise to purchase real estate in a different neighbourhood.

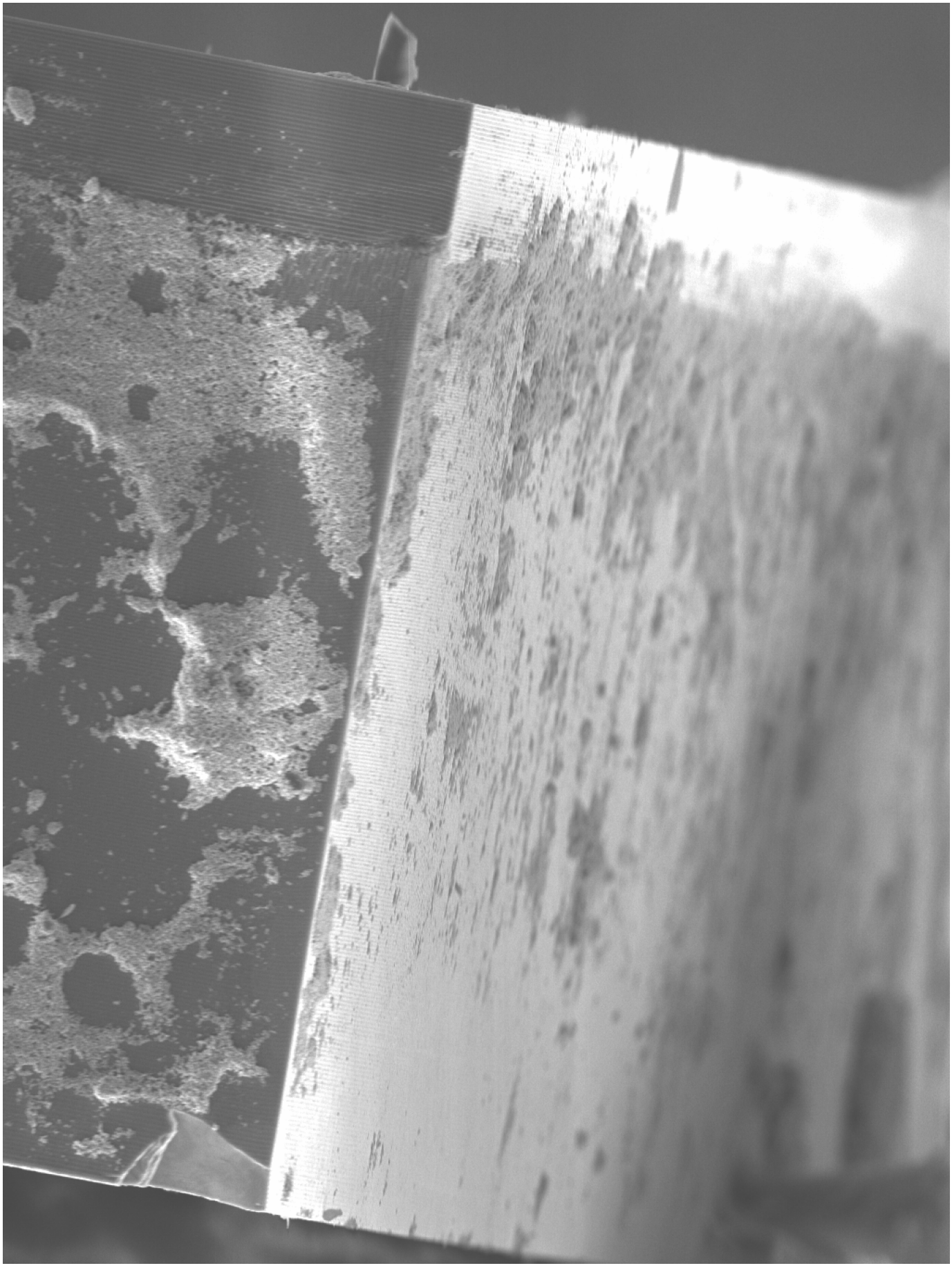


Scallops

25µm

This cross-section of a deep reactive ion etched wafer shows the ‘scallops’ of the Bosch process very clearly. A highly periodic ‘ribbed’ pattern is etched into the silicon, indicating that the etch process is approximately in the right regime in terms of balancing etching and deposition. Defects or erosion of the mask have resulted in different sections etching at different start times. The distance between each rib line is around 500nm.

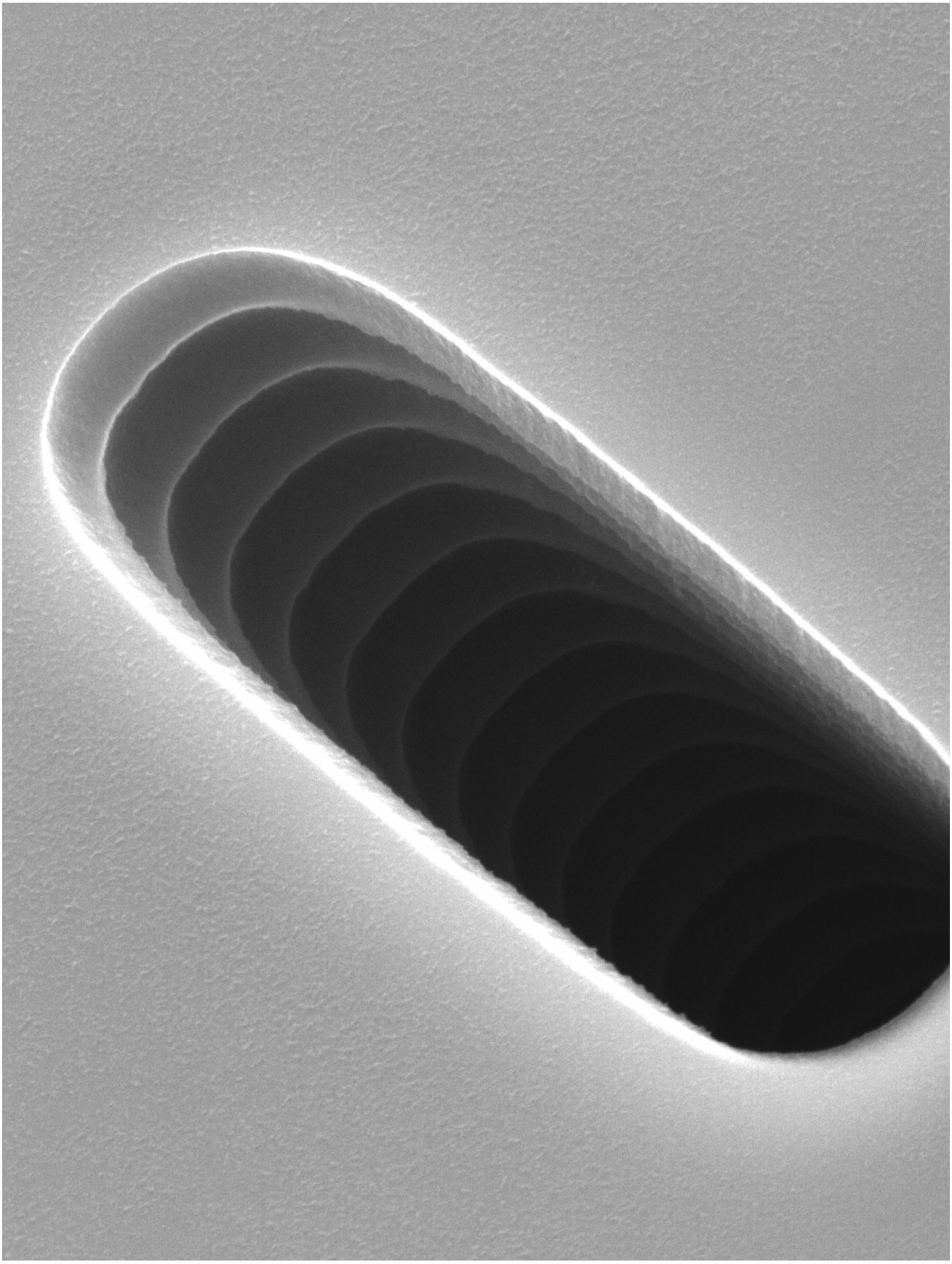
FEI Nova Nanosem 450



Let's Meet in the Middle

25µm

Here we see another scalloped silicon substrate, but this one is special in one way: it was etched from two sides, with the two etched trenches meeting to finally release the piece. The area where the two sections meet is clearly visible. This was done to enable the etching of small orifices in the silicon; which would not be possible if done in a single etch due to the high aspect ratio involved. The blobs of thermal paste on the substrate unfortunately detract slightly from the flawlessly scalloped sidewall.

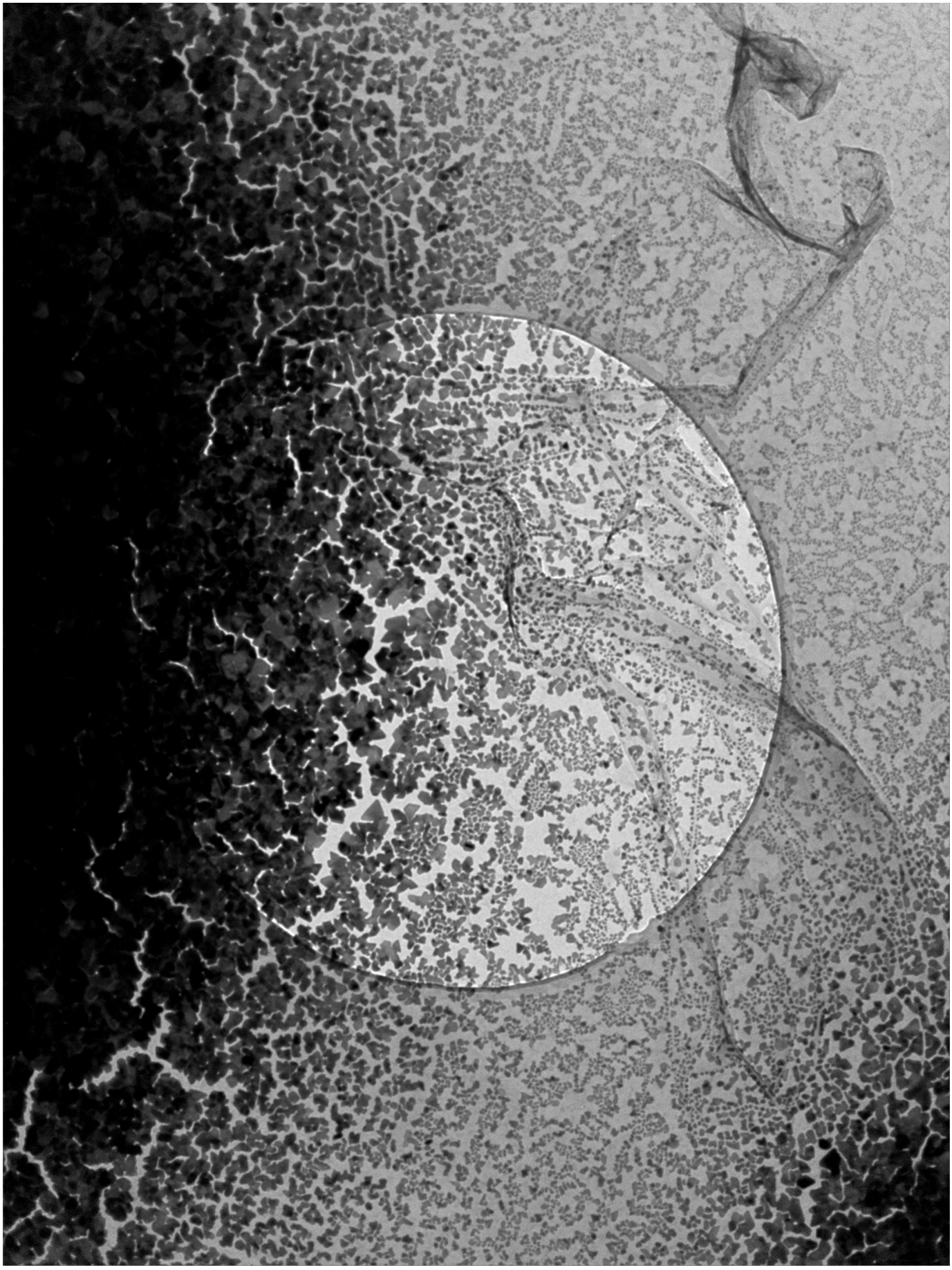


Scallops II

1µm

To make a reusable mold for carbon patterning, we used deep reactive ion etchting to make small blind holes into a silicon substrate. This high magnification image shows the scallops intrinsic to this process in greater detail. This project did not end up going anywhere due to lack of suitable evaporation materials. Nevertherless, far be it for me to pass up on an excuse to do some SEM imaging.

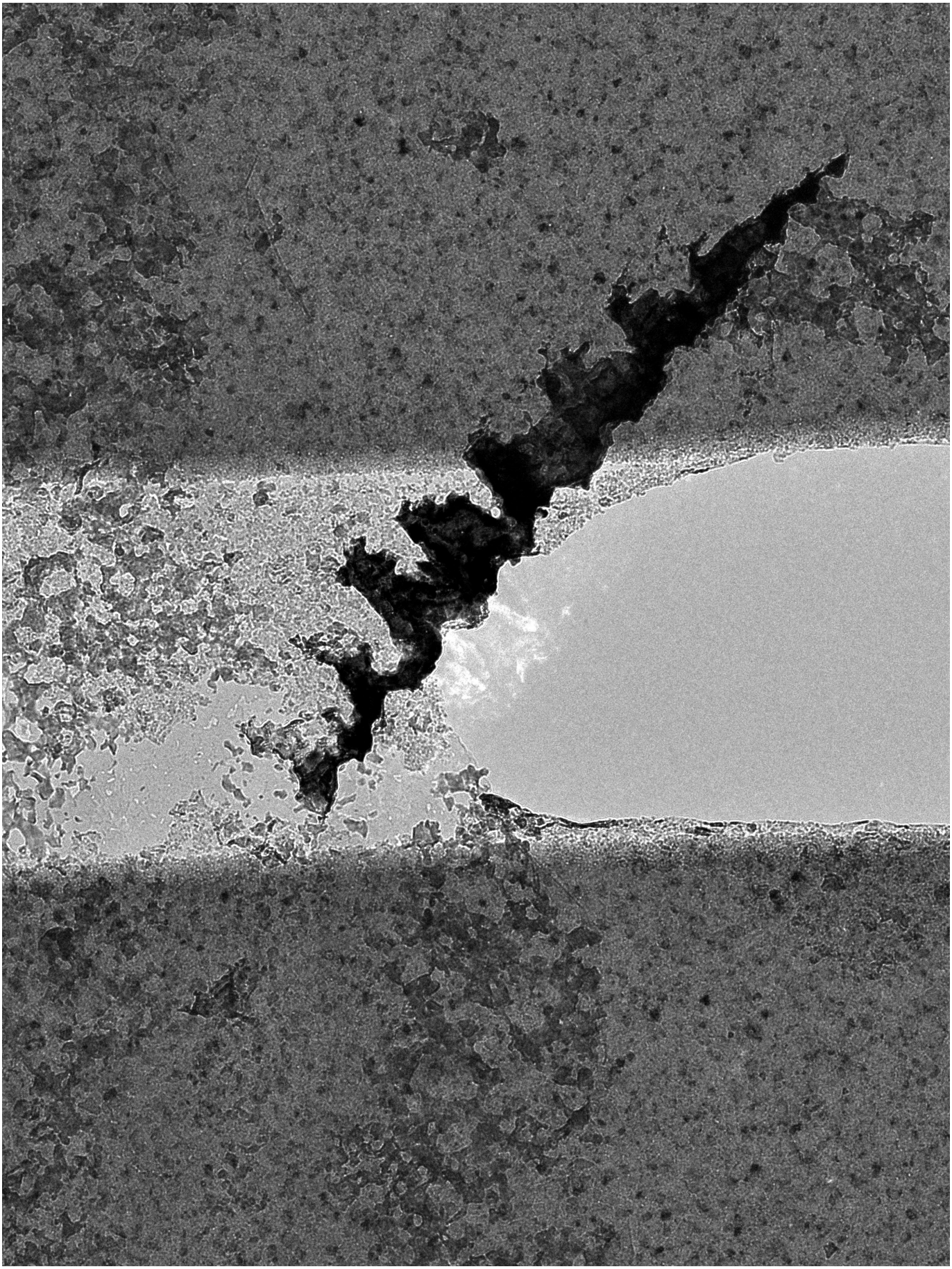
FEI Nova Nanosem 450



Gold Rush

300nm

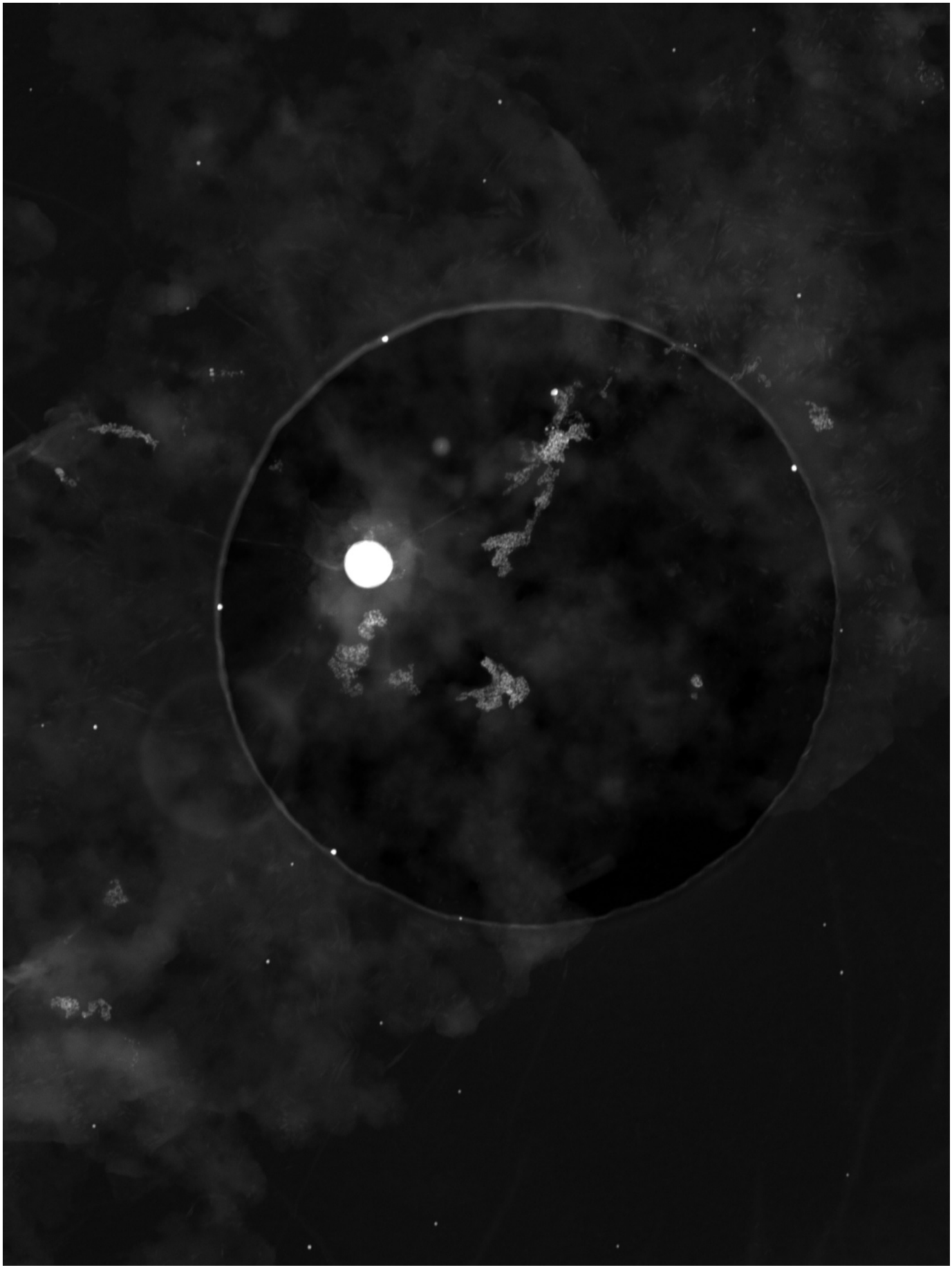
This image was recorded by Thomas Kock. This is a transmission electron microscope (TEM) image of the sample depicted in Inorganic 12. The gold dots visible in that image forms the black shape visible in the left of this image. When we set out to do this experiment, we failed to anticipate that once the hot gold atoms are evaporated, they will have high mobility. Rather than staying stuck to their original landing location, the atoms diffuse and form nanoscale (angular) gold crystals on top of the graphene membrane.



Orphaned Scallop

1µm

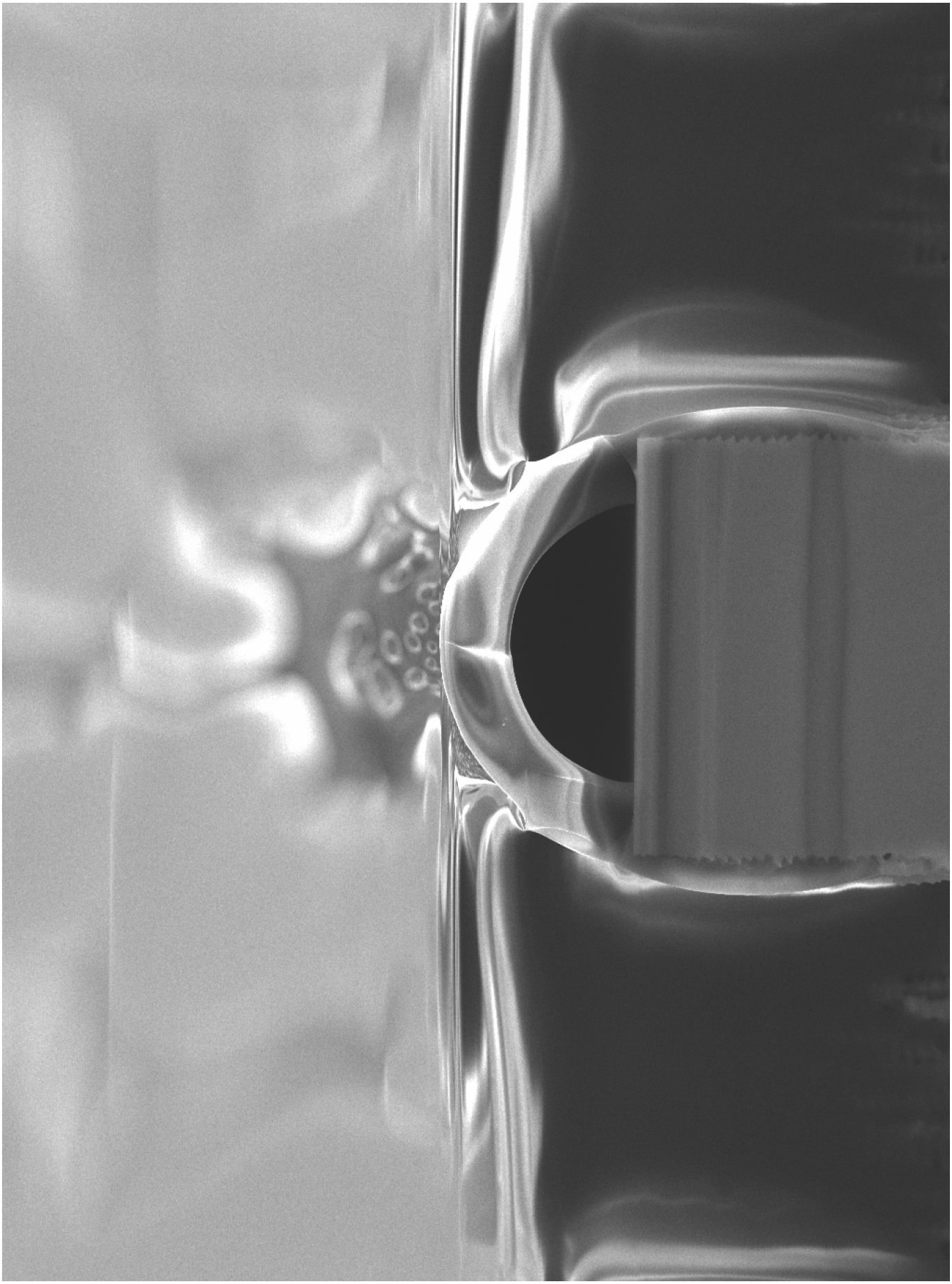
Under normal deep reactive ion etching conditions, the scallops form on the bulk silicon substrate, which is unlikely to break under normal handling conditions. Here, however, one of the scalloped sections of the sidewall seems to have ended up on the thin suspended aluminium oxide membranes of our chips. How did this little fellow end up fracturing of the bulk silicon substrate? It can't have been for the quality of the graphene, which is covered in some unidentified contamination.



Full Moon

300nm

To get to the bottom of strange spherical particle contamination on our graphene we turned to dark field scanning transmission electron microscopy. By mapping the element-specific x-ray emission, we found that our ‘full moon’ was likely zirconium or zirconia. This then lead to the next question: where is this zirconium coming from? I don’t think we were ever able to conclusively pinpoint the source. At least we can definitively conclude that the moon is not made out of cheese. Science advances one observation at a time.



Clairvoyance

10µm

In this image, we are looking at a cross-section of a silicon substrate after deep reactive ion etching. The photoresist mask used had poor adhesion the silicon and ended up forming this astonishingly perfect arch on the substrate. But the miracles do not end there: we are able to learn something about the side of the substrate that is not directly visible in the image! Charge buildup of the 'hidden' side is causing strange lensing to take place just above the arch, and we see a heavily distorted image of the hidden surface.

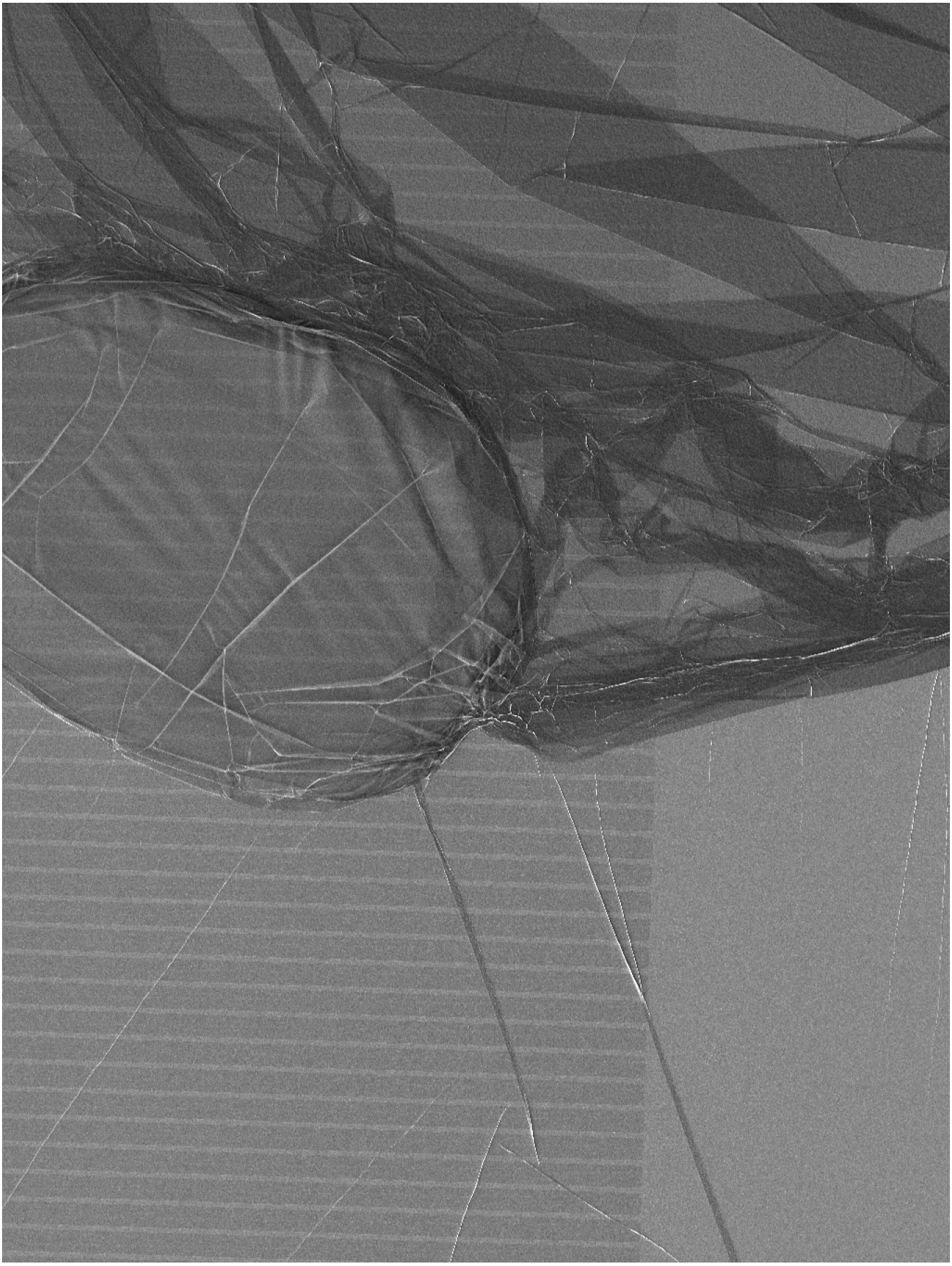
FEI Nova Nanosem 450

Clean Your Room

10µm

Microfabrication work is carried out in cleanroom environments to minimise the amount of particles that can land on and ruin devices. I am not sure if this is a dust particle, or some other kind of picked up contamination, but these were certainly too small to spot by eye. Despite their 'small' size, they dwarf all features on the device. The big dust particle is so large, that its charge buildup is causing electron lensing.

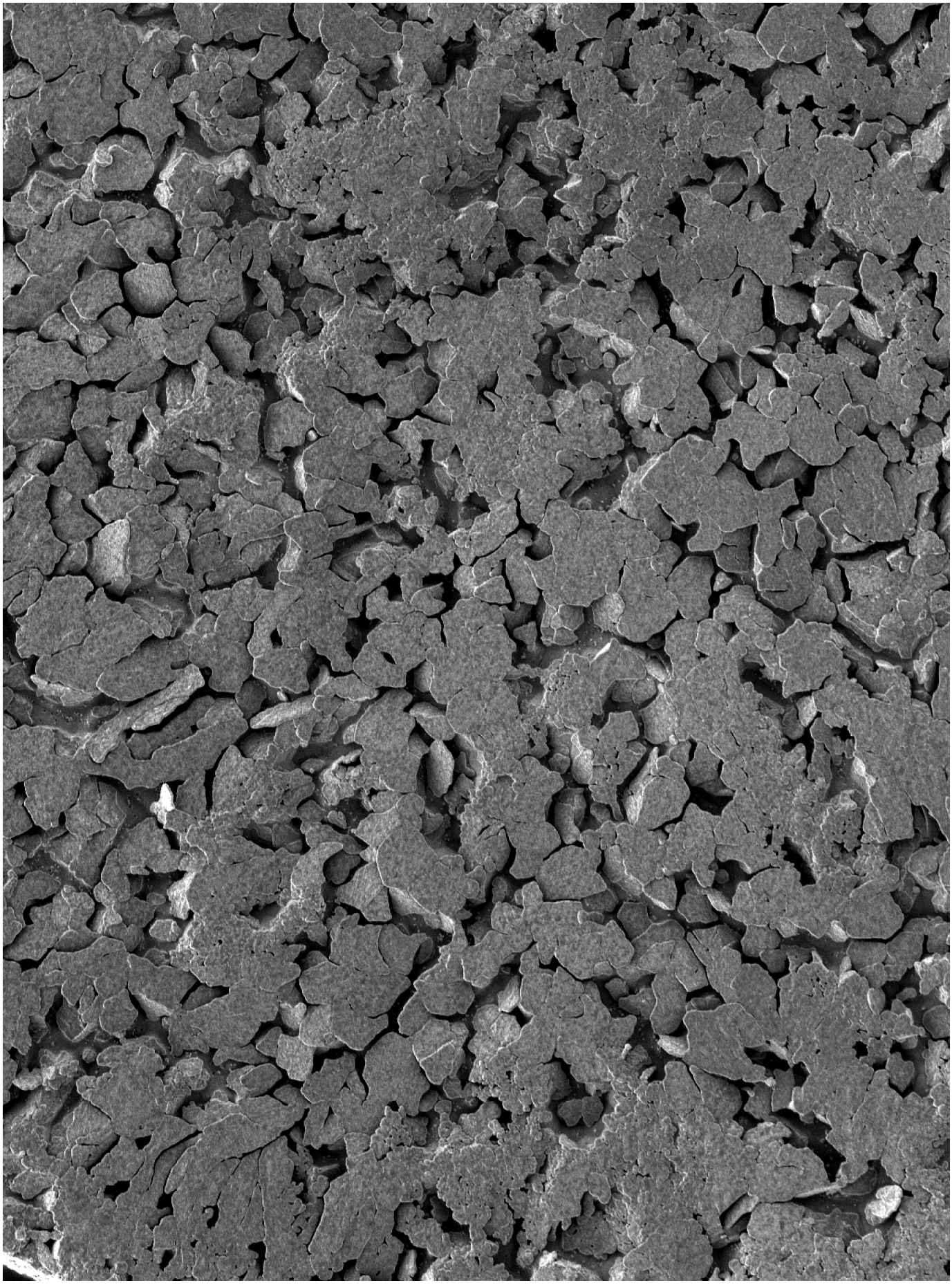
FEI Nova Nanosem 450



Graphene Origami

50µm

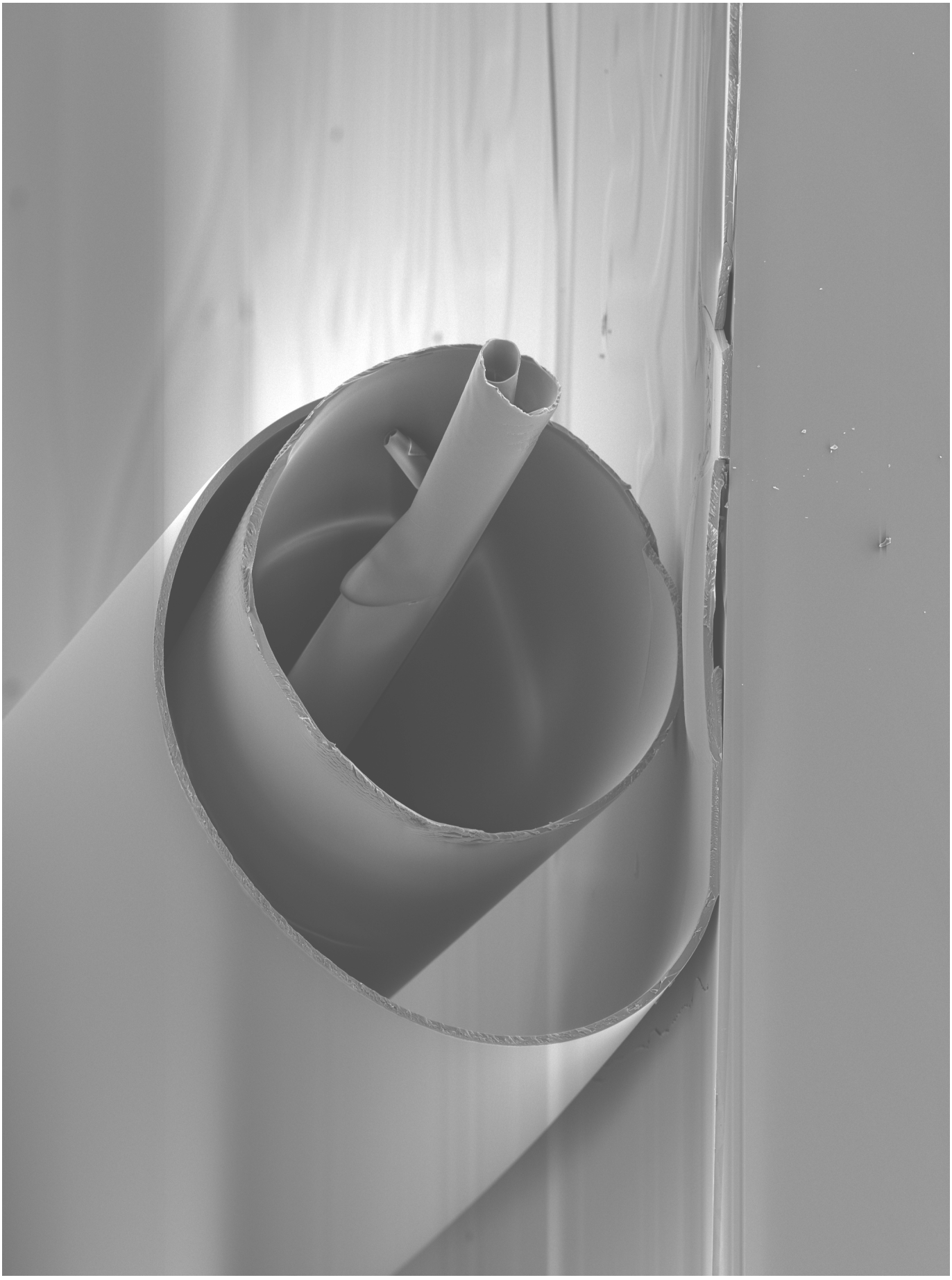
This image shows a location on a sample similar to Polymers 26. Unlike that image, here the graphene layer has ended up folding onto itself many times as the support was etched away and the substrate was dried. Needless to say, nature needs a bit more origami practice.



Shatterpoint

50µm

This highly fractured surface was formed after CVD of molybdenum on silicon with only it's native oxide layer. At high temperatures, the molybdenum is able to diffuse into and alloy with the silcion lattice, as seen in Inorganic 03. Unlike the nickel sample of Inorganic 01 & 02, there is no sign of pyramidal openings.



Cinnamon Stick

200µm

Polyimide is a high-temperature polymer that we tried to employ as microchannel capping layer. While other problems prevented its use, the polyimide has appreciable tensile stress after coating and curing. In this section of the wafer, a large section of polymimide sat on top of a PMGI layer, which was subsequently etched away. The polyimide was then able to freely curl up due its own tensile stress.

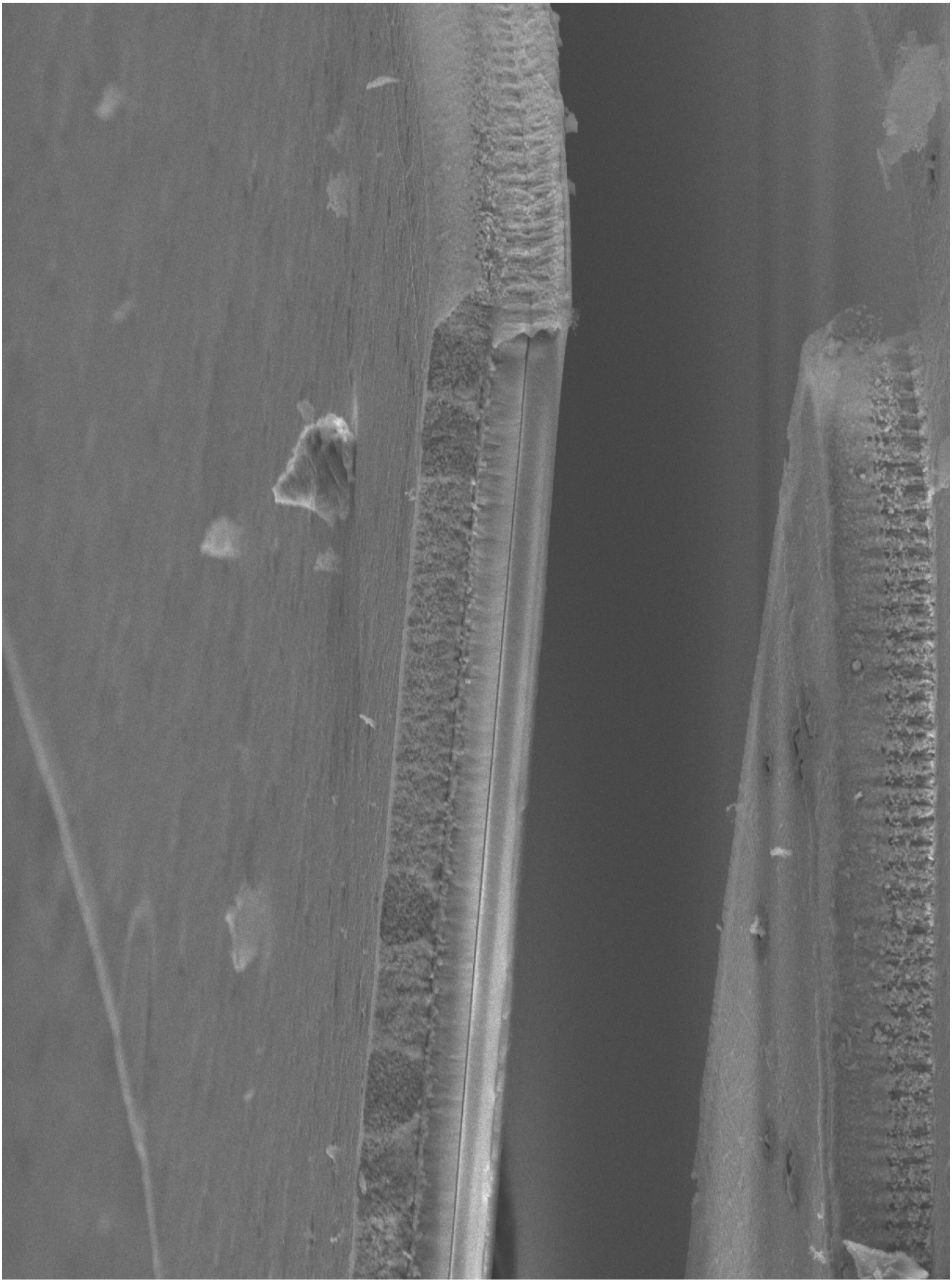


Cutting the Tension

3µm

Ultrathin membranes are also, as one might expect, ultra fragile. Aluminium oxide membranes as we used them are under tensile stress. If this tensile stress exceeds the yield stress or if other mechanical forces act on the membrane, it can fracture. Suspended membranes under tensile stress sometimes crack in this unusual wavey fashion. I'll take an interesting failure mode over a boring one.

Stack: Silicon | Silicon Dioxide | Aluminium Oxide | Silicon Dioxide | Aluminium Oxide



Archaeology

10µm

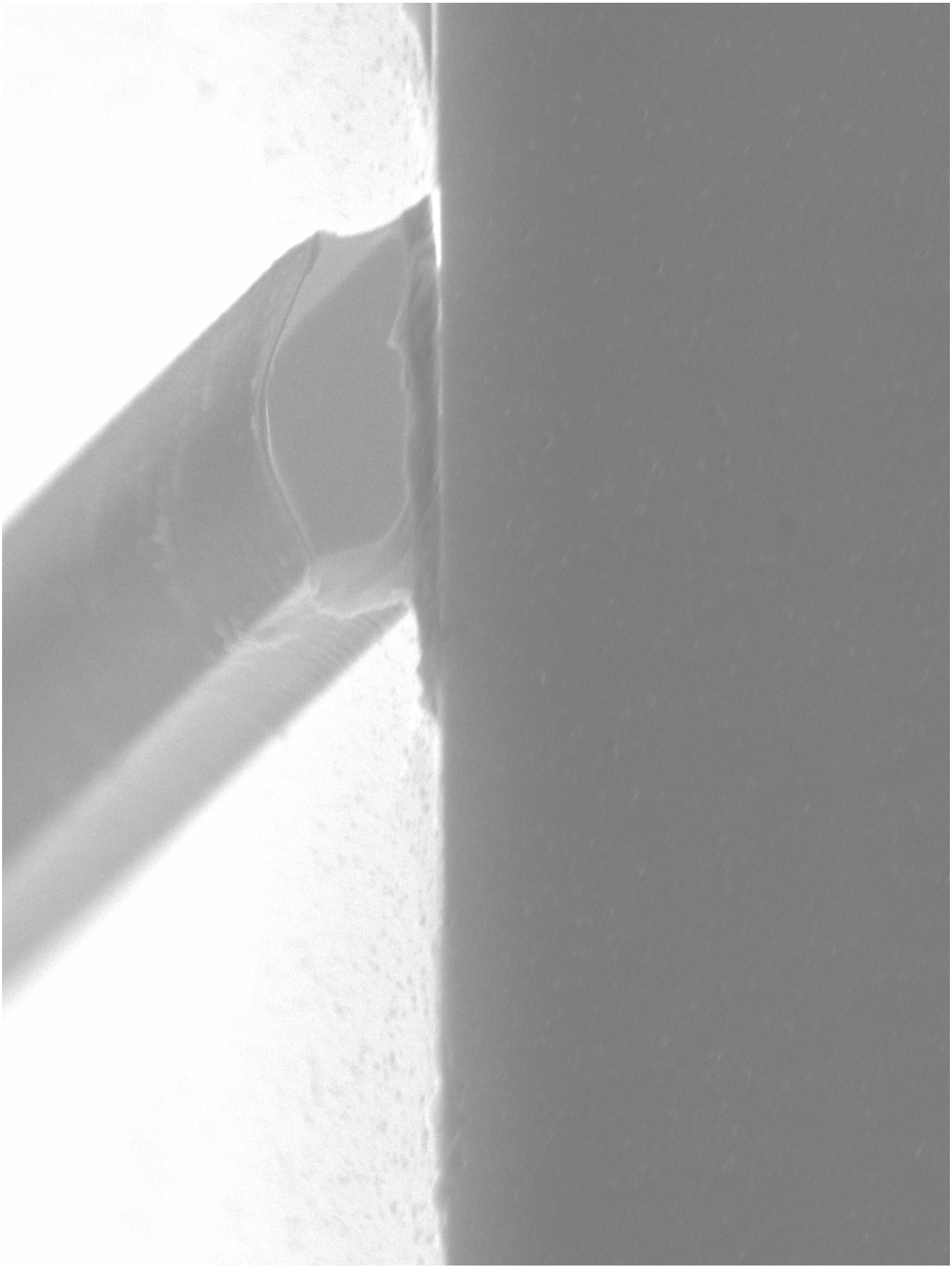
This layer sandwhich decided to delaminate after a hydrofluoric acid (HF) vapor treatment. In theory, the aluminium oxide should have prevented the vapor from etching the topmost aluminium oxide layer, but we can see that the sections on the side were almost entirely removed. On top of that, the layer stack decided to delaminate in its entirety from the surface, giving us a cross-section-like view of the stack history. The topmost silicon dioxide layer looks rather porous; likely due to contact with the HF vapor.

FEI Nova Nanosem 450

The Tunnel

1µm

Forming channels for liquid to flow through can be quite straightforward: simply pattern a line of sacrificial material, and thereafter deposit a thick layer on top to seal the system, and then remove the sacrificial layer. This concept is shown here for a small microchannel made using photoresist as a sacrificial layer. The real challenge begins when you need to make the encapsulating layer as thin as possible, and all materials must be optically transparent and have low autofluorescence. Then it becomes a tunnel with no end in sight.



The Tunnel II

2µm

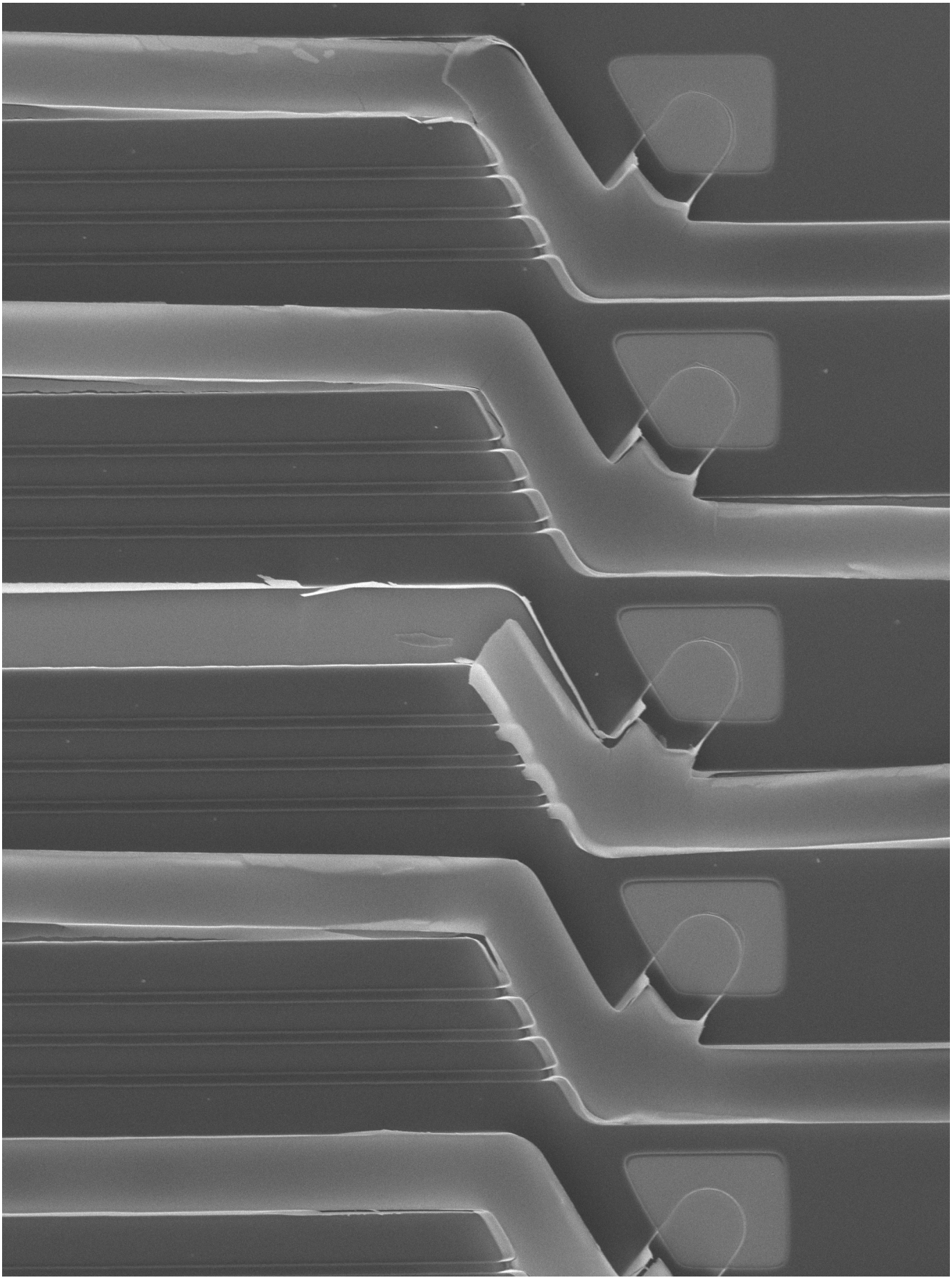
What if we take the same kind of microchannel as the previous image, and now remove only the topmost silicon carbide, leaving the 19nm of aluminium oxide? The answer, apparently, is an extremely fragile microchannel. The microchannel shown here still has a 200nm layer of polymer at the bottom that is insoluble in the solvent used to remove the bulk of the sacrificial photoresist. This microchannel is truly only held together by hopes and dreams (and possibly residual polymer).



Translucent

2µm

A simulation very similar to that on the neighbouring page: a 19nm aluminium oxide membrane that is held together only by residual polymer inside the channel and a tiny amount of intrinsic strength. This time, we can also see nanochannels run alongside the big microchannel, and we can see how the thin aluminium oxide membrane is electron translucent; we can see the outline of the channel through the vertical broken fragment. This property is essential for us, as we want to do electron imaging through these alumina layers.

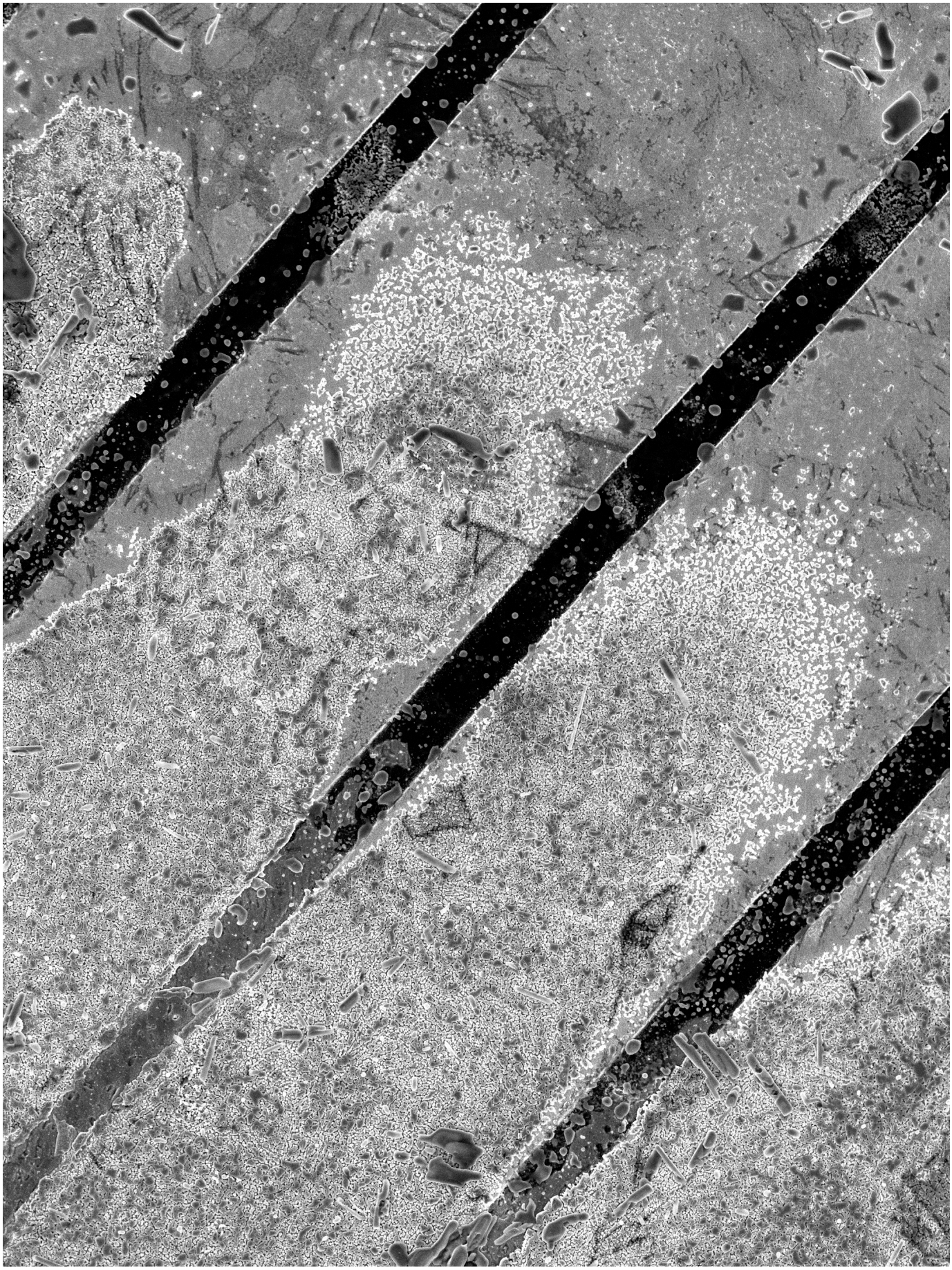


Delidded

10µm

In an older arrangement of our fluidic devices, the microchannels ran right along the nanochannels. In order to mechanically reinforce these channels, we added an additional silicon dioxide layer. After removing the sacrificial support polymer and drying the substrate, we found that the microchannels were still clearly broken. This was especially dissapointing as most of the channels are still in the original position, giving the impression that—just maybe—the strategy had worked. Never celebrate too early.

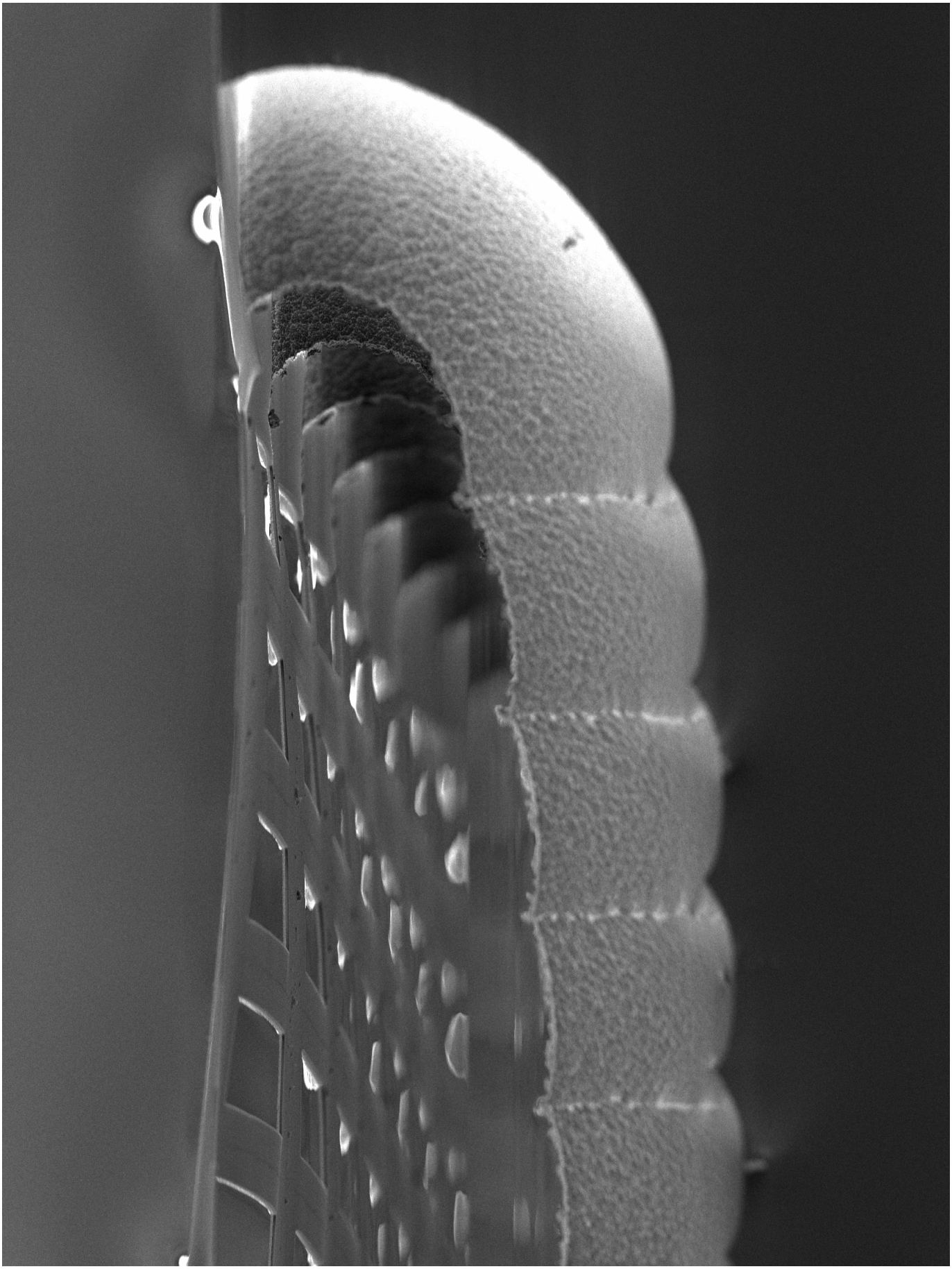
FEI Nova Nanosem 450



Crystals on Crystals

10µm

This sample was another clear example of the lesson also experienced in Inorganic 10: properly rinse your samples to get rid of residual salts. Ammonium persulphate was used to etch away the copper foil that the graphene was grown on, in order to suspend the graphene membranes. Gold strips would reinforce the graphene to prevent it from tearing. Under ideal conditions, the etchant should not have been able to get on top of the gold to begin with—but ideal conditions exist only in my dreams and diagrams.

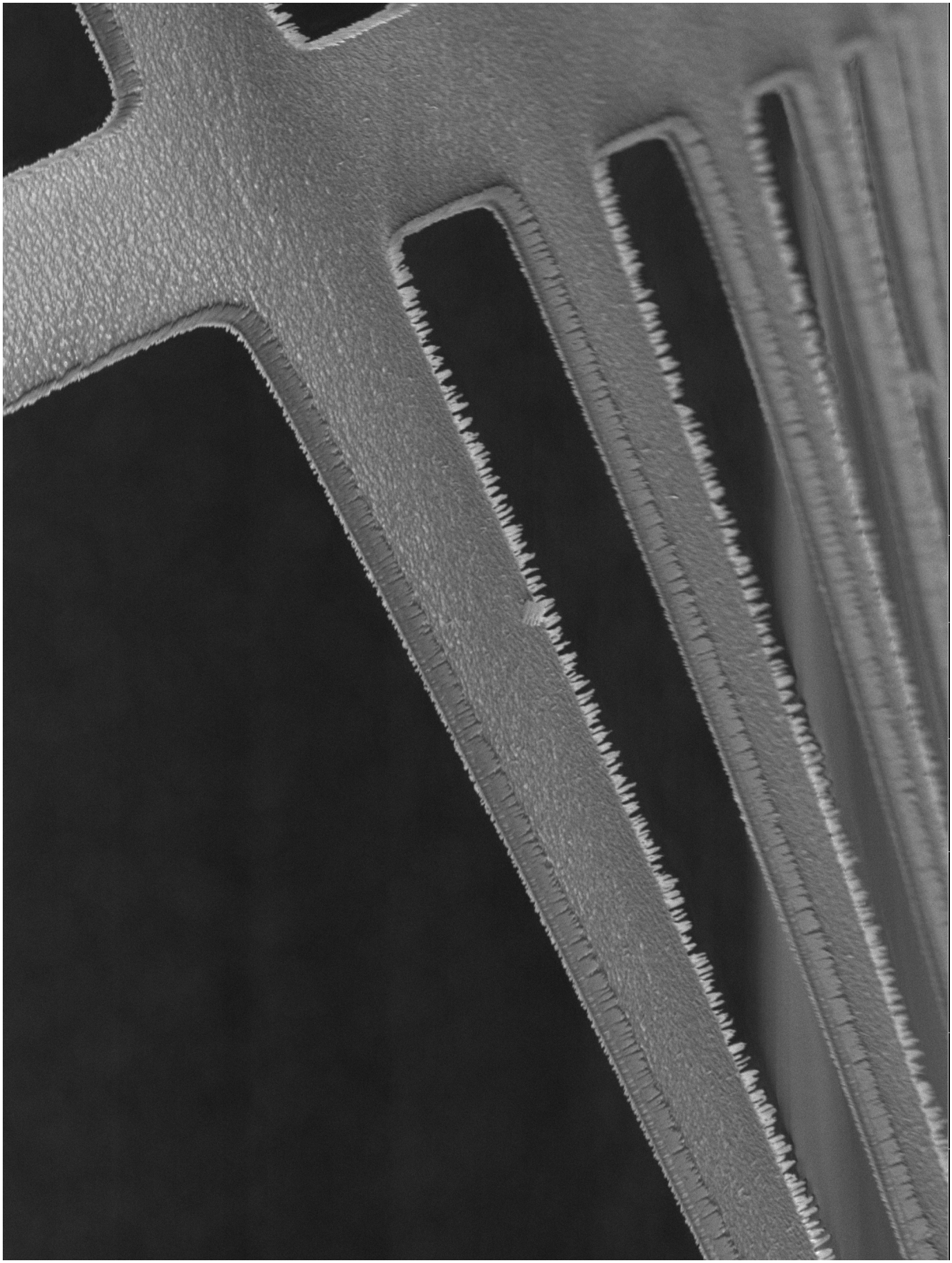


Bonus Cavity

40µm

The thin suspended membrane containing rectangular openings is a silicon dioxide layer that was supposed to be supported along most of its length. I did not, however, take any measures to prevent charge buildup at the bottom of the deep reactive ion etched trench (see Electron Microscopy 02). This meant that the silicon etch ended up continuing sideways, eventually removing all of the support between different windows. Instead of a supported silicion dioxide layer, a suspended membrane and a bonus cavity was formed.

FEI Nova Nanosem 450

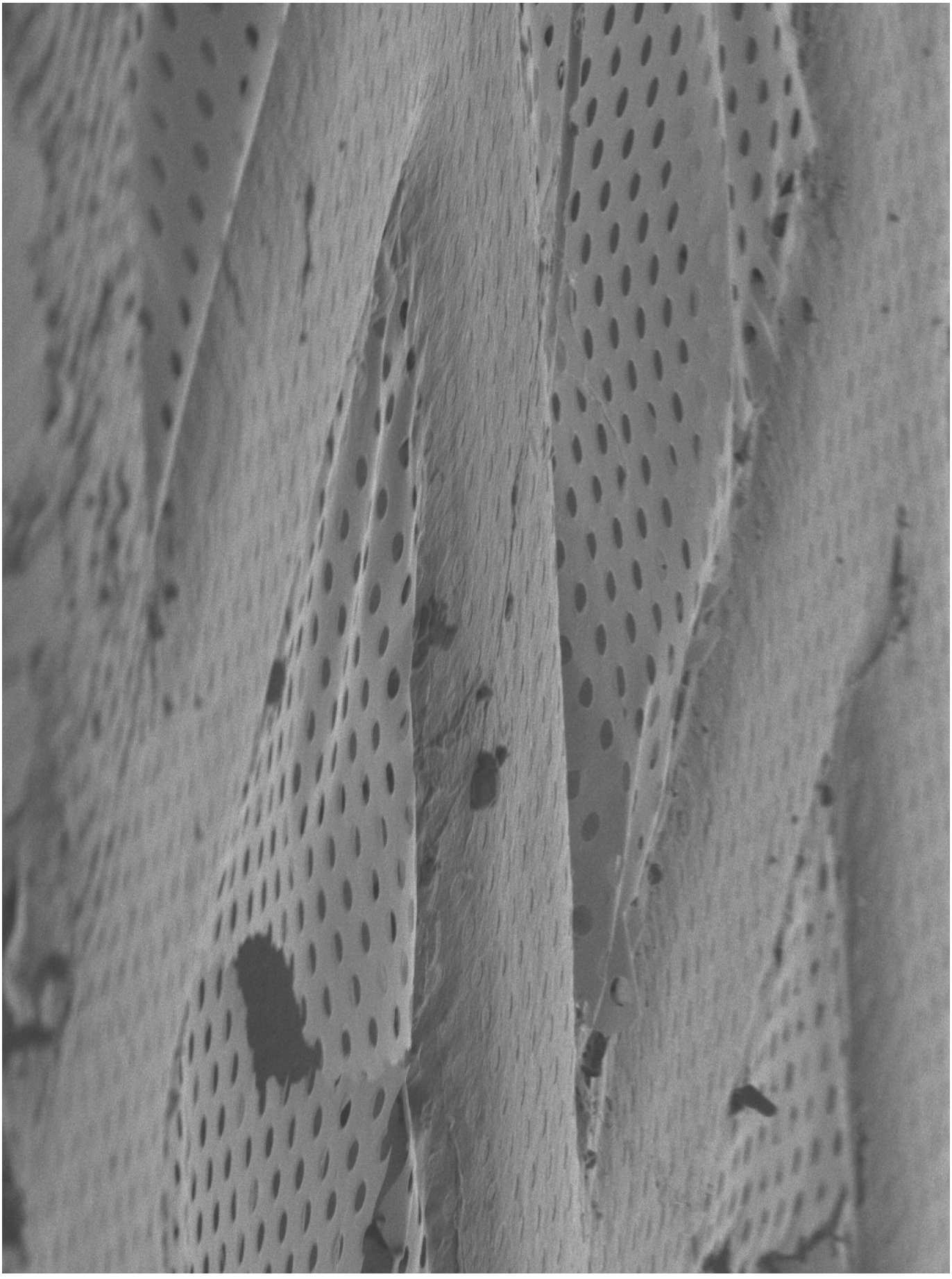


It's Cold Outside

10µm

This close up of the membrane also featured in the image on the left shows icicle-like structures sticking out from the membrane edges. These 'icicles' are likely made out of silicon dioxide that was redeposited on the etchmask's sidewalls. The silicon dioxide layer was etched in a separate process involving a large amount of kinetic etching. This kind of ion milling process is known to redeposit some of the removed materials on the sidewall of masks. I am not happy to admit it, but hydrofluoric acid does have its advantages.

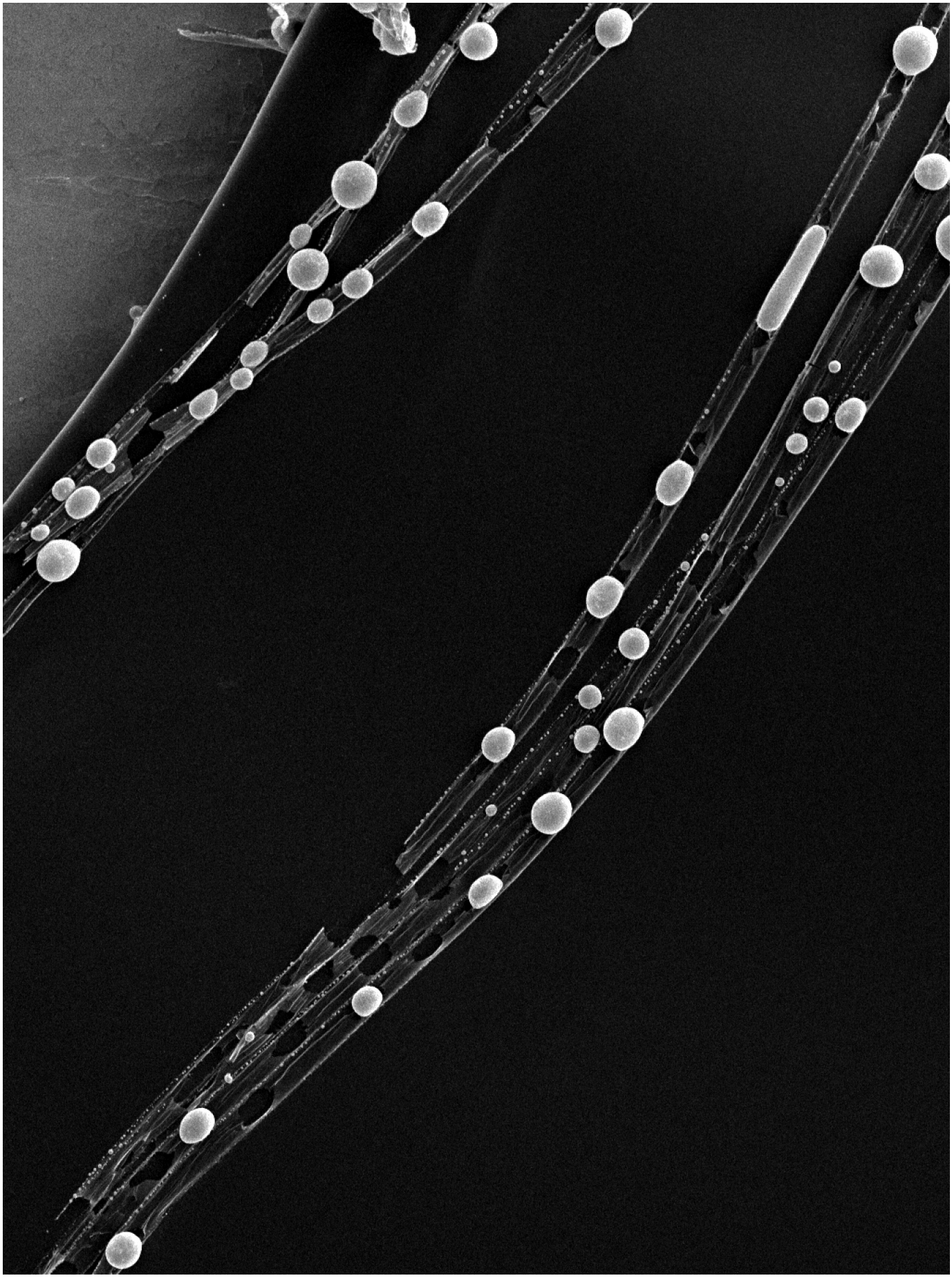
FEI Nova Nanosem 450



Stormy Seas

10µm

This is a standard copper TEM grid that would be used for protein structure determination using cryogenic-sample electron microscopy. I somehow ended up damaging the carbon films on this particular sample, making the membranes look particularly wrinkled.



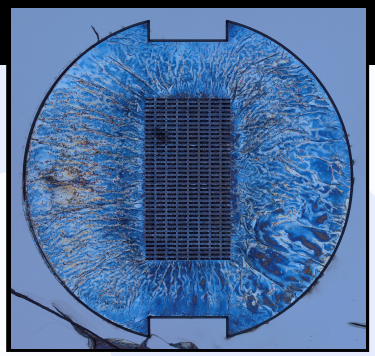
Surface Tension

10µm

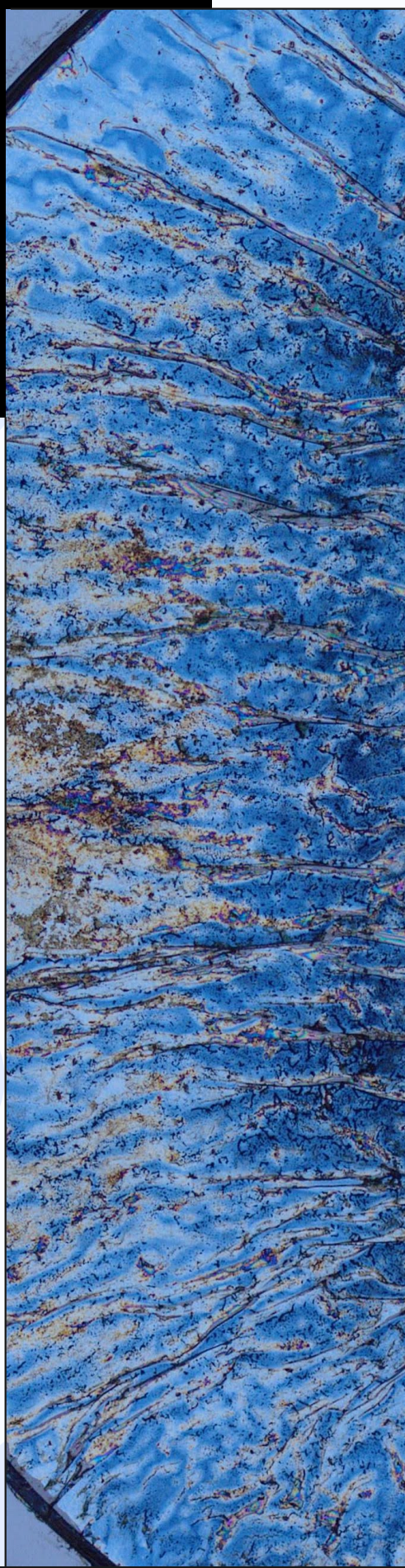
If you were wondering what would happen to Polymers 30 if you were to put the sample in a 930°C CVD process, you will find the answer here. The photoresist layer was completely reduced to a layer of ash-black carbon. The gold, however, does not like to wet this carbon layer and ended up forming small beads to minimise it's surface tension. Gold atoms' high mobility is also why a lot of cleanroom equipment do not allow or restrict exposed gold layers. Once gold contaminates the system, getting it out will be a nightmare.

FEI Nova Nanosem 450

The cover image shows a silicon substrate that was deep reactive ion etched all the way through the substrate using a photoresist mask. In this process, the photoresist is exposed to a plasma with fluorine-containing radicals. These radicals react with the resist, forming a thin 'skin' that will no longer dissolve in organic solvents. Here we see this thin 'skin' still attached to the silicon substrate, even after the soluble part of the resist mask was already removed in organic solvent. The wrinkled pattern shows various colours, which are caused by different thicknesses of residual resist or solvent still being trapped underneath this 'skin' layer. This chip was meant to be used for liquid-phase electron microscopy: and features ultra-thin membranes on the other (not visible) side. The rectangular array in the image is a series of holes in the substrate that are required to be able to image trough the chip. Looking at the complete image in the small inset, I can't shake the feeling that the chip is also looking back at the viewer...



Stack Silicon Photoresist



This short photobook contains micrographs (images recorded with a microscope) that were recorded during the research described in the PhD dissertation "Nanofluidic Liquid-Phase Electron Microscopy of Microtubule Dynamics: development of a next-generation LP-EM system", carried out at the TU Delft and Leiden University.

These images span a wide range of spatial sizes, from the milimeter-scale, where the features in the image would be just about visible by eye, to the nanometer scale, where even a light microscope is not powerful enough to produce an image.

Inside, you will find a wide range of materials, colours, and fabrication processes. When these processes did not yield the desired end products or otherwise failed, it would sometimes result in beautiful images - or landscapes appearing under the microscope. These microscopic landscapes have been compiled together in this short photobook with the intention of bringing this unseen small world to a larger audience.

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