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Webinar Transcript

Preparation of Polymer Samples for Microspectroscopy

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PRESENTER: Kristen D. Wiley

Kristen joined McCrone Associates in 2000, and contributes her special skills to the handling and manipulation of fine particles—sometimes as small as a few hundred nanometers. As a result of research conducted in association with Chicago's Brookfield Zoo, she has published several articles on animal hair microscopy and comparison. She has also contributed several sample characterizations and photomicrographs to the McCrone Atlas of Microscopic Particles. Kristen is also an instructor for Hooke College of Applied Sciences.

Introduction

Hello, my name is Charles Zona, and I'd like to thank everyone for attending today's webinar. Our topic today is the Preparation of Polymer Samples for Microspectroscopy, and it will be presented by Kristen Wiley and Heidi Talesky. But before we get started, I would like to talk a little bit about The McCrone Group, for those of you who aren't familiar with our services.

The McCrone Group consists of three divisions: Hooke College of Applied Sciences, McCrone Microscopes & Accessories, and McCrone Associates.

Hooke College of Applied Sciences—that's our education division, offers professional development courses in materials science, including instrument-based courses such as polarized light microscopy, scanning and transmission electron microscopy, micro-FTIR and Raman, along with special topics courses, such as gunshot residue analysis, specimen isolation and preparation, which we're going to hear a little bit about today, fiber identification, pharmaceutical contaminants, and a whole host of other courses.

Hooke College is also an academic partner with North Central College and Concordia University, offering 3+1 programs which result in a bachelor's degree in applied microscopy, or chemical microscopy.

For a complete listing of our professional development courses, or more information about our 3+1 programs, please visit our website.

McCrone Microscopes & Accessories is our instrument sales division. They are a preferred dealer for Nikon industrial microscopes and metrology systems, and also the national dealer for the JEOL NeoScope benchtop scanning electron microscopy system, and are a preferred national dealer for Linkam thermal stage systems, and many other microscopy-based laboratory tools and supplies.

McCrone Associates, the analytical division of The McCrone Group, is an A2LA accredited laboratory providing materials analysis services, specializing in solving industry's toughest problems, using light and electron microscopy, X-ray diffraction, Raman, FTIR, and other cutting-edge techniques and applications.

So for more information on any of the three divisions of The McCrone Group, please visit The McCrone Group website.

And now I'd like to introduce today's presenters: Kristen and Heidi are both senior cleanroom microscopists at McCrone Associates, with over 30 years of combined experience. They're also instructors for Hooke College of Applied Sciences where they teach specialized sample preparation techniques for polarized light microscopy, micro-FTIR and Raman,

Kristen demonstrates how to use a wedge slide to press a polymer sample.

and scanning and transmission electron microscopy. For those of you interested in attending their course this year, it will be offered October 6th through the 8th, and for more information about the course, you can visit the Hooke College website for more details.

At the end of the webinar, Kristen and Heidi will field questions from the audience, and you can type your questions into the chat box.

Today's webinar is being recorded, and will be available on The McCrone Group website, right under the webinars tab.

And now, I will hand the program over to Kristen Wiley.

Preparing Polymer Samples

Thanks so much, Chuck, and I just want to take a minute to thank everyone who has taken the time out of their busy schedules to join us here today. I'm really going to try to keep this short and sweet, and at the end, like Chuck mentioned, I'll answer any questions that anyone may have.

So I'm just going to jump right in, here. As most of you know, polymers can come in a variety of different forms, such as parts and components, they can make up layers of multi-layered films, or they could be packaging materials, such as these rubber stoppers and centrifuge tubes that you see here.

Today I'm going to go over two techniques that I use for mounting two different types of packaging materials for FTIR analysis in transmission.

We're going to take a look at rubber stoppers, which are a soft, elastomeric polymer. We're also going to take a look at a plastic shaving from a tube, which is a harder polymer.

Selection of the appropriate sampling method is crucial to achieving good results. There are several out there to choose from, and the two we're going to talk about today are, what we call here at McCrone, the micro cover glass method, and the glass wedge method.

The micro cover glass method requires small micro cover glasses as you see here, which are actually made from your standard large cover glasses. Most everybody has a large cover glass laying around, so you can make these yourself.

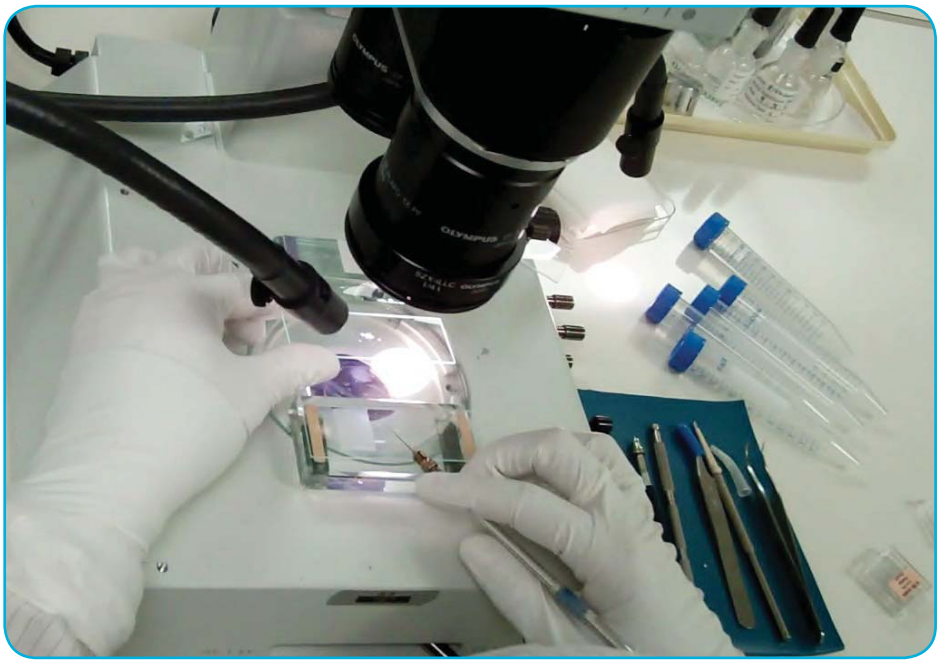
You do this by scoring one side of the large cover glass with the diamond scribe, in both the vertical and horizontal direction. This gives you the ability to make them as large or small as you need.

The glass wedge method requires the use of a glass slide, and we turn it into what we call a wedge. And again, everyone has slides laying around, so you can make these yourself. You make the wedge by scoring the corner of a glass slide with a carbide scribe, leaving an approximately 1 mm foot on one end, and then snapping it (the scored section) off. You just want to make sure that when you do the snapping, you snap away from yourself, and be careful so you don't cut yourself.

Some other micro tools that you might need when you're preparing small particles—small particles require small tools—so some other micro tools that you may need are a tungsten needle, a carbide scribe, curved forceps, and a substrate holder, such as this KBr holder as you see here.

The substrate I'm going to be working on today is a micro potassium bromide crystal, or micro KBr. We purchase it as a 1" x 1" crystal, and it can be cleaved in several different areas to make several smaller KBr crystals, as you see in that box here on the right.

So now I'm going to show you a video. This first one, this first video here, is going to cover the micro cover glass method. Right here, you see me, I'm transferring smaller pieces of my sample, with my tungsten needle, to my substrate slide, which has my micro KBr crystal on it. From the smaller pieces, using the side of my needle, I cut away even smaller pieces, which will be easier to work with and produce a much better spectra.
I'm going to now use my curved forceps to place a micro cover glass over my sample, and using a carbide scribe, I'm going to apply pressure to the top of the glass, and swirl the cover glass in a circular motion. By doing this, I'm breaking down the elastomeric properties and creating a very sticky residue. This residue should stick to the cover of the micro cover glass, allowing you to transfer it directly to the KBr.



Kristen Wiley has assembled her microtools and supplies, and begins to prepare her sample for microspectroscopy.

So now I'm going to come back in with my forceps, pick up the cover glass, and place it directly onto the KBr crystal. You can then apply pressure again, with your carbide scribe, to the top of the cover glass, and drag it away from your sample. This transfers the residue then to your KBr crystal, leaving a nice, thin film. I always come in with my needle—the side of my needle—and scribe a box around my sample, which makes it easier to relocate my sample once it's in the instrument.

Now the same technique is going to be shown here again, but from a side angle, so you can see what it looks like from the side.

I'm using my forceps to place the micro cover glass over my sample. I'm taking, now, my carbide scribe, applying pressure, and dragging it away from my sample. Now the micro cover glass can just be discarded. So, that was the micro cover glass method.

This next video here—we're going to take a look at the plastic, harder—the harder plastic pieces. So here I have the plastic shavings, and again I'm going to cut smaller pieces—this time, I'm using a 45° micro blade. And now I'm going to switch back to my tungsten needle to move these smaller pieces, and relocate them to a different area on my slide so I have room to manipulate it with my glass wedge. I'm really going to try to make sure that these smaller pieces fall between 200 μm to 150 μm; if not even less than that.

I'm now going to place my glass wedge directly over my particle, and with the carbide scribe, I'm applying pressure to the foot. I'm going to keep applying pressure while moving the wedge back and forth, until I obtain a nice, thin film.

Now here again, the technique's going to be reviewed again, but this time from the side. You can see I'm applying pressure with the carbide scribe, and my opposite hand is moving the wedge back and forth, until my sample is thin enough.

Now if you're lucky, sometimes your sample sticks to the wedge itself, and in that case, the wedge can then just be transferred to the micro KBr crystal, and you can apply pressure with the carbide scribe, transferring your sample to your KBr crystal.

Some polymers, you'll have to cut smaller pieces from your flattened piece, which I'm doing here with my tungsten needle, which actually is giving me a little bit of a difficulty so I'm going to switch to my 45° micro blade. I'm then going to press it again with a micro cover glass, so my piece is actually thin enough to obtain a spectra.

Now this thin piece can then be transferred to the micro KBr with my tungsten needle. It has to now be pressed out again, so I'm going to cover it with the micro cover glass, apply pressure with my carbide scribe, and drag it off as I did in the previous video.

Some polymers tend to stick a little bit to the micro cover glass, so that step may have to be repeated a few times. And again, I'm just coming in here with my needle and scribing a box around so I can relocate it into the instrument.

This last step actually shows me transferring the KBr crystal to the substrate holder. This slide can then be placed directly into the instrument.

Now some of you may be wondering what the results are of these two samples that I just mounted, and that's to be continued…so if you'd like to see how those two samples ended up, you can tune in on September 3rd for Andrea Champagne's and Mary Stellmack's webinar on Techniques for Obtaining Infrared Spectra. And again, that will be held on September 3rd.
So at this time, if anyone has any questions about anything you've seen here today, I'd be more than happy to answer any questions someone may have.

Questions & Answers

CZ:
Thanks, Kristen. That's really interesting stuff. Yeah, if you have any questions, please type them into the chat box there, and we'll start taking a look at those. I think we have one here: "What magnification do you work at?"

KW:
Oh, that's a good question. Everyone finds their "happy" magnification. I prefer to work roughly between 40X and 60X. And I should also mention that everything that you've seen here was done on a stereomicroscope (not a polarized light microscope), which obviously has a much higher working distance, and you can get your hands under there. So, again, I prefer to work between 40X and 60X, but that may depend on each individual.

CZ:
Okay. Let's see; "What size are the tungsten needles?"

KW:
The tungsten needles we have, they are made here, in house, and they're made using a .5 mm diameter tungsten wire. And we sharpen that with sodium nitrite to obtain the sharpness that we need for our individual samples.

CZ:
Okay. "What other techniques do you cover in the course?"

KW:
The course covers a variety of sample techniques. We cover mounting polymers, paints, fibers, hairs; all for FTIR and SEM analysis. So again, it's not just for FTIR like we covered today, but also for SEM and EDS. I actually strongly recommend that students should bring in their own samples, so that we can help them obtain the results that they actually need once they get back to their lab.

CZ:
Boy, the questions are really streaming in now. "Can you do the analysis of the sample mounted on a microscope slide vs. the KBr?"

KW:
I don't believe so, I think glass actually causes too much interference, so you need something that can be invisible to IR. So that's why we prefer the micro KBr crystal.

CZ:
Let's see here…I can't scroll through these fast enough! "Does the method accept an official method?" Is there some sort of official method for this?

KW:
Is it accepted as an official method? That—maybe—I don't know if the person who wrote that question, if they're referring to something like SOP related, or an ASTM method, I'm not sure about that one. I'd have to jot that down and contact you directly to find out specifically more what you're inquiring.

CZ:
"Can we get a copy of this presentation?" Yeah, it will be available, again, on our website under the Webinars tab, so the complete presentation and the transcript actually will be available as well.

CZ:
I just put up my information. You can contact me at kwiley@mccrone.com. I'd be happy to answer any questions that anyone may have. If I don't know the answer, I'd be more than happy to find out for you. Are there any more questions?

CZ:
"I missed how you mounted the KBr to the special holder. Is that two-sided tape?"

KW:
The KBr holder is actually…the KBr holder itself is something that McCrone Microscopes & Accessories sells. It's a special holder that's hollow in the middle, and there's a very light adhesive around that hollow opening, so the edges of the KBr crystal are attached to that adhesive, but leaving the center of the KBr, where your samples are prepped, hollow, so the laser can actually come up through the KBr crystal and hit your sample. I hope that answers your question.

CZ:
"How do you flatten high-density or rigid polymers?"

KW:
The same way, it just takes a little bit more time, a little bit more patience. The glass wedge actually works really great for that. You might have to originally cut a smaller piece. I think the key there is always start small, and right when you think you have small, go smaller. So it takes a lot of time, a lot of patience, but you can break that down and break it up, and you'll see, eventually, what first appears hard will eventually become soft. Once it becomes soft, you can get it flat, and then you can get your spectrum.

CZ:
There were a couple of questions like that. Very similar in nature. "Do you sell tungsten needles and other sample prep tools?" Yes, we do at McCrone Microscopes & Accessories. They sell needles that are already sharpened. I believe they're fine, medium and coarse, so you can definitely get a lot of these supplies from McCrone Microscopes.

KW:
They also sell the micro cover glasses, which are really handy, but unfortunately, the micro KBr crystals—we've had bad luck trying to ship them in the mail with humidity, because it is KBr. Depending on the humidity, how much moisture is in the air, it can actually damage the surface of the KBr. So we haven't had much luck shipping those. But I encourage everybody to try making them. They're very easy to make, and we actually make them and cover that in my three-day class.

CZ:
Let's see…"Do you use separate KBr discs for each sample analyzed? If not, how is it best to prevent contamination as samples are pressed into the disc itself?"

KW:
That's a good question. I think the best way to answer that is, it depends on how confident you are in your sample preparation. Someone that is probably just starting out, you probably don't want to put more than three or four samples on your KBr; you want to keep them spaced out. I sometimes use the side of my needle to kind of divide up my crystal, so I know what sample is in what area. Once you get really good at it, you can probably end up with 10 or 20 samples on one KBr crystal, and it also depends on the size of your sample. Sometimes, if we're really pushing the threshold of small, I could put—if my particles are 30 μm to 40 μm each, I could put 10 to 20 on one KBr crystal, but if you have a larger sample, like I was saying, 100-150 μm, then you probably want to keep to just a couple of samples per KBr.

CZ:
Next question, "Do you make your micro KBr from your regular cuttings in the lab?"

KW:
We actually purchase a 1" x 1" crystal, and from that, we just use a razor blade to and cleave the crystal in several different areas. One crystal we can probably get roughly between 40 and 50 micro crystals. If they are kind of, like you're saying, irregular, if I am understanding your question, if they are a little irregular and rough, we use a very fine grit sandpaper to sand it down so it's nice and even, and a flat surface, and then we polish it with a little bit of water on a clean wipe, and if you repeat that motion a few times, you should get a nice, smooth surface.

CZ:
There's several questions here I think regarding infrared—questions like ATR versus transmission, and things of that nature, which I think we'll address next week with Andrea and Mary—some of those questions. Let's see… "Does pressing the sample induce strain in the sample and affect the IR spectra?"

KW:
Not that I'm aware of. I think if the sample's small enough, there's really no strain in the sample, so your spectra would be the same. So, there wouldn't be…you have it thin enough. The only time it would become problematic is if you have several polymers that you're pressing together, like if it's a multi-layer film, and they're each something different (each layer), and you press that in all one big…as one big particle, that's going to cause a problem; but there are ways and methods to actually separate out individual layers if you have a multi-layer film—which is a whole 'nother webinar.

CZ:
A couple more questions here… "If you have a metal particle, can you use the same method?"

KW:
Metal particles cannot be pressed out transparent, so usually metals cannot be run in transmission for FTIR. I would suggest metals being done on an SEM or an EDS.

CZ:
And another question here: "What about plastics with high carbon black content?"

KW:
That's interesting. If you press them—again, if it looks jet black—you start larger, and you keep cutting smaller and smaller, and eventually you press it so thin, you're getting light through it. A kind of rule of thumb: if you get light through it, you can get a spectra. So it has to be thin enough where, yes, you might still be seeing tiny little carbon spheres in there, but there's enough of the clear area to where you would obtain a spectra.

Left to Right: Nicole Groshon and Kristen Wiley perform sample preparation and particle isolation in McCrone Associates' ISO Class 5 cleanrooms, which are certified to ISO 14644 standards.

CZ:
Let's see here… "Does your class cover embedding of multiple laminated polymers?"

KW:
We don't cover embedding, but we definitely have different techniques that we cover. We actually do hand-sectioning and cross-sectioning of the multi-layered films, or multi-laminate films; I don't know if that is specifically what you're interested in, but in the class, again, I would encourage you to bring your samples with because we do cover quite a bit of cross-sectioning in the class.

Conclusion

CZ:
Well, okay then, I think that kind of wraps it up for the questions, and again, I'd like to thank everbody for attending and hopefully we'll see you next time, next week, actually, for our webinar on Techniques for Obtaining Infrared Spectra, with Andrea Champagne and Mary Stellmack. Thank you very much.

KW:
Thank you!