Graphite-Molten Salt Interactions
Dr. Nidia Gallego, ORNL, USA

Berta
Welcome everyone to the NextGen 4 International Forum Webinar presentation. Today's presentation on Graphite-Molten Salt Interactions will be presented by Dr. Nidia Gallego. Doing the introduction today, Dr. Patricia Paviet, Dr. Paviet is the group leader of the Radiological Materials Group at Pacific Northwest National Laboratory. She's also the National Technical Director of the Molten Salt Reactor program for the US Department of Energy. She's also the Chair of the Gen IV International Forum Education and Training Working Group responsible for bringing you these wonderful GIF webinars. Without any further delay, I give you Patricia.

Dr. Patricia Paviet
Thank you very much, doctor. Good morning, good evening everyone. It's a pleasure to have with us Dr. Nidia Gallego. She is a Distinguished Research Scientist in the Physical Science Directorate at Oak Ridge National Laboratory in the United States. She earned her Masters and Ph.D. in Materials Science and Engineering from Clemson University, and she joined Oak Ridge National Laboratory in December 2000. Her research interests include, among others, physical and chemical properties of carbon materials, effect of neutron irradiation on graphite and carbon materials for use on space power systems. Currently, Nidia is the Oak Ridge National Lab Technical Lead for the graphite activities for both the Gas-Cooled Reactor and Molten Salt Reactor campaigns funded by the US Department of Energy Advanced Reactor Technologies Program. And she's also the Task Lead for Production of Carbon-Bonded Carbon Fiber components as part of the Radioisotope Power Systems Program, which is funded by NASA. Without any delay, Nidia, I give you the floor. Thank you very, very much for presenting this webinar. Thank you, Nidia.

Dr. Nidia Gallego
Thank you. Thank you, Patricia. Thank you, Berta. Can everybody hear me okay? I hope.

Dr. Patricia Paviet
Yes, we can.

Dr. Nidia Gallego
Thank you, Patricia. It is a pleasure for me to be here this morning, this afternoon, presenting this seminar. I wanted to first of all remind everybody that this was intended to be the second on a series of two webinars related to graphite materials. Last month, Dr. Will Windes

from Idaho presented a webinar titled 'Overview of Nuclear Graphite R&D in Support of Advanced Reactors.' In that discussion, he covered in great detail graphite irradiation behavior, oxidation, and some more background on the various reactor concepts that utilizes graphite. He also talked a little bit about the myths about whether graphite burns or not and mentioned that the second webinar this month will be talking on the graphite molten salt interactions.

I should point out that our recording of that presentation, as Berta mentioned, is also available on the GIF website alongside his slides. Before I go into the technical details of my presentation, I would like to acknowledge the ORNL contributors to the work that are going to be presented. Primarily, I would like to acknowledge contributions from Cristian Contescu, Jisue Moon, Yuxuan Zhang, Jim Keiser, Adam Willoughby, Dino, Jun Qu, Xin He, who had contributed significantly to the work that again I'll be presenting here. We also collaborate not just with this core team, but other ORNL members and within the campaign and outside. We had collaborators at Idaho, other academia and industry organizations.

Financial funding from the work that I will be presenting, it comes from the US Department of Energy-Nuclear Energy Advanced Reactor Technology Program and some other work that I will be presenting also use resources at the High Flux Isotope Reactor, which is a US DOE Office of Science User Facility. Very grateful to this wonderful team of collaborators that I have.

Before I go into details about a couple slides from Will's seminar last month, just to put everybody into context, we'll give a good, a nice overview of what is called now the nuclear renaissance and mention the various reactor design concepts that are part of Gen IV. There is quite a few of them that utilizes graphite at the core, not only as a moderator, but also some structural roles in there. He also went to details that some of these high temperature reactors that use graphite at the core can either be cooled by gas in the case of the helium gas-cooled reactors, or could be utilizing a molten salt as a coolant or also as a carrier for the fuel.

With that a quick overview, outline of what I will be talking today. I will go back and do a quick review of the nuclear graphite materials and the microstructure emphasizing the microstructural features that are of interest to the topic of this, which is the interactions with molten salts. Then I will be covering in detail two main topics that are critical for the ASME qualification of graphite for use in high temperature reactors, which is understanding the graphite soil intrusion into graphite and the effect that this may have in graphite properties and understanding graphite wear behavior. This goes along with graphite and erosion. We've been tackling wear behavior and hoping to get into erosion topics following this. I will have a slide on summary and ongoing activities.

With that just a quick – most people I'm assuming here that are present are familiar with the graphite macrostructure. Graphite crystal structure is a hexagonal compact, AB layering. However, the actual manufacturer of graphite or blocks of graphite is a more complex structure. We got filler particles or cooked particles in most cases. We had a binder material that puts together this and then this is molded using various techniques, can be extruded, vibromolded, isopressed, and during that process, and the cycle of heat treatments. During that process, there is significant amount of pores that are entrapped off developed due to the outgassing of the binder. Most manufactured graphite or nuclear grade graphite will have about 20% porosity. This is one fifth of a block of graphite, its actual pore structure.

Why do I emphasize this? Why is this porosity in graphite important? This is important because this porosity defines the irradiation behavior of graphite. The very typical and unique behavior that graphite has with irradiation where graphite has a negative dimension of change, there is a shrinkage on graphite as the neutron dose increases. Until there is a point where that, what we call accommodation porosity and the amount of defects that are being generated due to the radiation reaches a minimum. And then there is a turnaround point where graphite will start to expand again. Then it comes and cross over to the original dimensions. And then, in theory, it will continue to expand and will go above the original dimensional.

This is important because this typical behavior is going to be a function of the graphite grade. I will explain later what I mean by graphite grade. But that graphite grade is linked to a porosity, a typical porosity to that grade that is going to be defined by pore size and distribution. That turnaround point here in these dimensional vessels, neutron, those will determine the lifetime of the graphite in the reactor. This, again, is going to be a function of grade. And it's also going to be a function of temperature. This porosity in graphite always also is going to lead to external surfaces or edges. In graphite, we're going to have basal planes and we're going to have edge sites. The edge sites are going to be the reactive sites from graphite. And as you can imagine, when you have pores of different sizes, we're going to have different amount of edge sizes, the surface area. Therefore their oxidation rates are also going to correlate significantly with what the type of porosity or the size of the porosity in that graphite grade. There are different oxidation rates depending on your graphite grade.

Again, and this is very important when we're talking about gas-cooled reactors, because, as you may know, you know, helium is not 100% inert. It may be traced amongst the impurities in the helium. And then that needs to be accounted for to understand the oxidation rates and also in the case of oxygenated scenarios, it's important to understand how that graphite will be oxidizing.

But when it refers to molten salt, those pores might mean that salt may intrude into the pores. As salt intrude into the pores, that may also be affecting the graphite properties, whether it be mechanical or thermal. There may be also edge sites that may be susceptible to the interaction with the various components of the molten salt. This edge side may also be potential size for either tritium retention or reaction with other off gases in there.

I think I mentioned several times that when we talk about nuclear graphite, we talk about grades. There's a significant amount of graphite grades available there, even though they all meet the requirements for purity and be all close to 100% pure carbon, but there are a quite significant wide range of graphite grades. The main difference for the purpose of this seminar that differentiate these grades that I want to emphasize is that these grades will have porosity that comes in different shapes and sizes and connectivity.

The table below will list – it's not comprehensive, but it's a summary of some of the more relevant graphite grades that are currently being produced. H-451 is a historical grade, but we have the NBG-18 or -17, PCEA, IG-110 from the various manufacturers; either from US, Germany, France, or Japan. There is classification on class here. It is from the ASTM standard. Basically, defines the grain size of the graphite based on the size of the filler particles that were utilized at the beginning.

This grain size, together with this forming process, again can be extruded, vibromolded or isomolded is going to define that network of pores. One way to characterize that pore network is to do mercury intrusion. Again, this mercury is ideal for characterizing pores that are on the micron size.

Here, this graph on the right shows the mercury intrusion data for 12 graphite grades. As you can see, the profiles of that cumulative volume or mercury pressure. We have different patterns in here. We can separate these into two groups. We have the fine grain graphite that shows a very sharp uptake. Once this threshold pressure is achieved, you don't see any mercury intruded into the pore structured at these lower pressures. But once you achieve t:]his, a given pressure that is typical or specific for each graphite grade, we see this significant uptake. Again, this is a sign or indication that, again, first of all, we have very small pores, so mercury is not waiting [ph] in graphite. We have to overcome that force to not wanting to go into the pores. But once we reach the pressure that let us intrude into the pores, we'll see that the pores are highly interconnected. Then basically what we do is saturate the graphite because most of the pores are connected.

Contrary to these, we see the median and large grain graphites, where we see a larger distribution, a wider distribution of the pore sizes. These graphite grades will have pore sizes that are tens of microns in size. We see that there's mercury intrusion for very low pressures, and we see that continuing over this wider pressure range. This is the whole complete graph for those twelve graphite grades.

With this data, we can apply Washburn equation and the model that the Washburn equation use in pore sizes and port geometry, and derive the pore size distribution based on mercury intrusion. That is in this graph here.

As we can see, we can observe here two patterns. The fine-grain graphite shows a very narrow pore size distribution, very sharp and narrow distribution, basically telling us that most of the porosity is within this narrow pore size. The sizes, the average pore diameter for this graphite grade, like I mentioned before, it's under few microns. Complementing that, the median and large grain graphites will have a broader pore size distribution. And again, we have pores that will be tens of microns and in some cases, a few larger pores than that.

How do we go from mercury intrusion into salt intrusion? There is ASTM standard within the community. A few years back, there was this development. It is not a standard test method, it's a standard guideline, and it primarily defines the apparatus and the procedure for exposing graphite specimens to molten salts at high pressures, at temperature. The ASTM also introduces two quantification parameters, the D0 and Dt. These parameters are just a ratio or fraction of the either open total porosity, a fraction of the open porosity or total porosity that was coverage or filled with. Based on the weight changes of the sample before and after the salt intrusion.

Unfortunately, the guide does not specify any sample geometry or size, and it does not specify any equilibrium conditions. Under most recent revision of this standard guide, a note was added just to bring the attention to the user that the user needs to be cognizant and aware that literature values that may bring up these D parameters into it needed to be taken into consideration that there may be differences in sample geometry, equilibrium conditions that may lead to different D parameters.

The one deficiency that in my opinion the guide has is that the guide does not make any reference about where the salt, or makes reference to the fact that the salt may be distributed or uptaken by the sample in different conditions. Therefore, may be distributed differently across the sample geometry. Again, all that because of what I spent a few minutes explaining the variations on both sides and distribution.

At ORNL, we have developed our capabilities to expose graphite samples to molten FLiNaK. Our system was developed and approved to operate at temperatures up to 750 degrees Celsius and we can go up to pressures of 10 bar. Our sample holder and everything that is in contact with the salt. It is made out of graphite, just to avoid additional effects or corrosion effects because of metal alloy components as opposed to salt. So, everything that touches the salt in this case it is graphite. But like I mentioned, most of that effort has been focused not on just measuring and quantifying the uptake as a weight percentage or as a D parameter, but on trying to understand, once samples are exposed to molten salt, how that salt is distributed within the graphite.

For that, we've been doing neutron imaging. We have here at ORNL, the neutron imaging beam line at HFIR. We have taken samples. We did this original proof of principle with samples that were supposed to FLiNaK at 5 bar, at 750 degree Celsius, 5 bars for 12 hours. The samples were a square cross section of 10 by 10 millimeters and 15 millimeters in height. We have these samples available when [Unclear] proof of principle time became available. We loaded these samples into the sample holders that are basically just on aluminum cans that are sealed. Once we had exposed the samples to the molten FLiNaK, everything has been stored in a glovebox to avoid moisture uptake. Those soils, as most of you may know are highly hydrophilic. Everything again is loaded into a glovebox, maintaining an inert environment. We take this to a bin line. We have our sample holder here, and then we do our radiography and imaging. The holder will rotate the sample can, allowing for a 360 [imaging] of the sample. This image overlaid here basically gives you like a slide half of that radiography of those samples. In this case, we have these four graphite grades here that will represent different pore sizes.

After we’ve done the imaging, we take this and we're able to do a 3D reconstruction. We had our blocks here. I should probably mention that our neutron imaging basically is measuring the attenuation coefficient. This color scale here, pure graphite will have an attenuation coefficient close to 0.3. Green alone will be an indication that it's just mostly graphite in their carbon. The FLiNaK attenuation coefficient of the various components, primarily contributing from the lithium in the salt will have an attenuation coefficient of close to 0.5. The more red you see, the more indication of salt being present in that volume.

I guess I should have pointed out here that our image resolution was only 75. Therefore, as you can remember from those pore sizes, this is much larger than the pore size and those fine grain graphites. We'll be getting kind of [Unclear] volume in that box, so volume that we get. But still, I think as I show, give us very good information on the salt distribution.

We had taken this 3D reconstructed image and gone and do sections or extracted planes from various distances from the top surface, just so that we can understand how that salt is distributed; not only from the outer surfaces, but also from the top bottom.

So we have this graph in here. The first column in here is going to represent the plane closer to the outer surface. We decided to take these a little – just 0.5 millimeter away from the surface because of their own artifacts on the edges. But again, this represents the plane closer to the top surface. Then we continue to extract planes that are getting away from that until we get to this 7.5, which is the halfway of our sample geometry. And so this particular column will be representing that salt intrusion that is unidirectional from the four sides in here. Again, we do have, because of the square cross section, maybe some artifact on the corners of the sample. But I guess to point out here, and again the scale here is neutron attenuation. The more red means closer to more presence of lithium. But a significant difference we see between this PCEA, which is a medium and large grain extruded. We got large grain pores in here. Compared to that, these four grades in here that are fine grain graphites where we see that the salt is more concentrated on the edges. Finally, this last one, which is CGB. This is a historical grade that was used in the emissary experiment, that it had cell surfaces. And we can see there is not significant intrusion. What we have done with this is taking the physical characteristics of the graphite sample before the intrusion and the amount of salt uptaking and then normalization. We had translating this neutron coefficient, neutron attenuation coefficient maps into what I call here a coverage map. So we took this last plane here halfway to the height of the sample, and so normalization on that, and we translated that attenuation coefficient into coverage from 0 to 1. And as you can see, that middle plane, again, this. This follows very similar to attenuation. It's just a different way to look at the numbers.

We also calculated the salt coverage percentage, which is basically just an integration of the area under this line profile that it was taking halfway in that plane. I will point out that you may notice that up to this point, I have not mentioned any weight percentages uptaken. I done that on purpose because I didn't want to bias anybody with this half more or less percentage. We wanted to make sure that we understood more where the salt is going and how that profile is happening, how deep from the outer surface the salt was penetrating. But from this area under the curve of percentage, we can see that, for example, these two are very similar. We have 20 and 19; some errors in here, but are curved under the area. If I were to correlate that with the percentage of salt uptake, their trend, the trends are very similar. But you can see that 20% coverage is distributed very differently in the PCEA versus the NBG-18. ETU-10, for example, has a little higher coverage, but that is a little more concentrated on the edges, versus the PCEA, which seems to have a little less, but is distributed again over the whole cross section.

Additional things that those results that I just presented have been put together into a paper that is currently in the review. If you're interested in more details or want to go and review this, look into this paper. I hope it will be published pretty soon.

One thing that I pointed out when I mentioned the ASTM is that we didn't have guidance on sample geometry or size, and also, we didn't have guidance on equilibration conditions. Up to that point, we have been doing intrusion for about 12 hours. We decided to look into, is this a fully equilibrated situation or is that salt uptake is going to change with time?

We went back, applied for additional beam time. We got additional neutron imaging time. This time we moved to from the square cross sections to eliminate those cornice effects to cylindrical samples. We had samples that were 10 millimeters in diameter by 20 millimeters high. We were able to also do the imaging of the sample prior to the salt intrusion. So we have a true representation of that sample. We had to get averages. We just have that image. So we were able to subtract that from the sample after the intrusion. We did intrusion in this particular time at 3 bar 750, and we did the 12 hours, which was our standard up to this point. Then we kept samples for up to 2 weeks at these same conditions. Here, this is just a brief summary of two graphite grades, a fine grain graphite IG-110 and a larger grain graphite, NBG 18.

This is how the imaging of the sample prior to salt intrusion looks like. Here again, we cannot resolve individual pores because of the resolution and the pore sizes here being much smaller than the resolution that we have. But NBG-18, we can see that we can resolve on individual pores again due to the broader pore size distribution and the presence of relatively large pores on NBG-18. And then we see that at the 12 hours, there is no salt intrusion in IG-110. And after 2 weeks, we don't see any on the IG-110 either. But we see that on the NBG-18, we see some partial salt intrusion in here, but we see a lot more or significantly more after the 12 weeks.

With this data, again, now that we have the baseline from the sample power through the intrusion, we're able to do 3D reconstructions. And this is a reconstruction of the salt after the intrusion. We can see the spots where there may be some salt in the surfaces, and we can go over the volume and subtract the original image prior to intrusion and just leave behind the areas where that represents the presence of soil. Again, this is demonstrated how powerful this neutron imaging is for this particular situation and this great contrast that we have between graphite and the FLiNaK.

But beyond measuring this and imaging, we want to be able to predict salt intrusion based on fundamental properties of the soil and the graphite. Again, I mentioned the Washburn equation before. When it comes to when we utilize that is used for mercury intrusion, the Washburn equation uses fundamental properties of the salt, the temperature conditions, the surface tension, but it also takes into consideration not just the pore size, but the contact angle of that fluid and the graphite surface.

Again, surface tension, wetting angle, so all the effort at ORNL within the campaign is to better understand that wetting angles, contact angle. We had recently commissioned a high temperature contact angle measurement system. Basically, it's a cantilever system. We load our sample in here. We had a furnace, we covered here. We pull vacuum several times to make sure we have an inert environment. We flow argon. We bring up the furnace to temperature. Again, we have a camera here. We get a window. We get a light here. Then we get a detector where the image projects. And then we can observe the transition from solid to fluid and the changes in contact angle. Measure the contact angle and the changes of contact angle of the temperature range that we're doing or over the time frames that we're looking at. Just to give you an idea, here is the graphite sample. We are utilizing graphite, this, that are about

10 millimeters in diameter, couple millimeters in thickness. And this here is the salt. We're pelletizing the salt just to make sure that we have a defined geometry at the beginning to help [on the melt] process. This salt pellet is about 3 millimeters in diameter and that is about 8 milligrams of salt in here. Just to point out the melting point of FLiNaK is about 454. I got a video here that is just – again, it starts at 400, close to the melting point. We can see temperature increasing. We're getting close to that melt temperature. We see after that melt temperature is reached. You know that that salt goes from solid to liquid and it coalesces into this droplet. The contact angle here is above 90, it is about 130, in the range of 130. That is indication of non-wetting. So up to this point, the salt is not wetting the graphite surface. But as we see this temperature increasing here and as we approach the 700 and beyond, we see that that contact angle, this angle here is changing with the temperature and is decreasing. We see it getting very close to the 90. Indication that starting to wet. We also see the volume of that sphere changing slightly. It's also indicating that some of that salt, again, is going into the pores. And again, contact can go below 90 where the salt went from non-wetting to wetting.

We are doing this. We are looking at various variables, not just the graphite grade. This particular video that I show here is for graphite IG-110. We're doing this for the same graphite grades that I mentioned before. The twelve grades with different pro size distributions. To see if there's any effect, we're preparing samples with different surface finish. We're hoping to also do different surface pretreatments, maybe on the surface, to see if there is any change. Potentially looking at changing the environment in here to see if that will have an effect. Again, mostly on the surface tension of the salt, but how much that will affect the weighting of that. One thing also, we'll be looking at whether that contact angle will change if it's kept at a given temperature over a long period of time.

The second topic that I mentioned that I'll be talking is understanding wear behavior of graphite. Again, some of the direct composites, we have pebbles that we move that are moving into the molten salt or will have salt flowing through graphite channels. Wear and erosion are things that are important to understand. We had started to look at erosion, but don't have any data that's a little more challenging property or issue to address. But we have been looking and doing some proof of principles or initial studies on trying to understand wear off graphite immersed in a molten salt.

This is the original setup that we use. It was available at ORNL, had been used for some solar programs. They've been looking at some chloride salts in there, looking at [solar salts] corrosion and wear of [solar salts] and chloride salts. We have access to it. To do some proof of principle, we have these graphite pebbles that were provided to us by one of our industrial collaborators. This system is a pin on this kind of geometry. So, from these pebbles, we machine these graphite pins. The surface of the pin will have the curvature, maintain the curvature of this pebble. Then we did the initial studies on a 316L stainless steel. We loaded the salt in a glovebox, we sealed it, we brought it into this bell jar, brought down the furnace, and flow argon, and then pull out the cap, brought out the pin, and did our work testing.

We initially compared the same geometry, the pin on and the stainless steel, flowing just argon, and then we added the molten FLiNaK. This table here just basically summarizes the differences between the wear volume or whether a test was utilized in argon or we have molten FLiNaK present. The main takeaway here is that when we have what we call a dried environment or inert environment, in this case [that] flow in argon, the wear volume on the stainless steel was negative, which basically is an indication of deposition material, deposition from the graphite pin into the stainless steel, which is expected at these conditions. But when we added the molten FLiNaK, we see that our volume goes from negative to positive, meaning there is an erosion or wear in the stainless steel. Again, there is the presence of salt, there is corrosion, there is not only wear, but there is also corrosion of the stainless seal. There is a combined tribocorrosion effect in here.

Based on this and knowing that we can use the setup that we have, we proceeded to have a more controlled set of experiments. The second set of experiments that we did, we moved from the 316L to a 316H. And that conversations with some of our industrial collaborators they recommended that we look at 316H instead of the L. This is our matrix. Basically, we did two temperatures 650 and 550. We did three sliding speeds, rotation speeds in here. We went from 1 millimeters per second, 10 millimeters per second, and 100 millimeters per second. We kept the duration of the test constant and that's just to have the corrosion part constant in here. The time of the exposure time of the 316 stainless steel to the molten salt was constant. Then we added this other condition that we call here flooded or starved. That came out from when we were doing these experiments at the highest sliding speed, this rotation. We noted that at this very fast rotation speeds, the amount of salt that we had initially put in here that was covering the interface was climbing over the sides of the cup. And therefore this interface wasn't fully covered or immersed in the salt. We went back and did some more controlling measurements here at the lowest sliding speed where we reduced the amount of salt to just have a film or similar conditions to where we have here when the rotation was very fast.

Briefly, I'll go to some of the takeaways of the results. This graph here compares effect of temperature. I think a little bit as expected as we increased the temperature from 550 to 650, we see that the wear volume and the wear rate of both the graphite pin and the 316H flat increases. Again, as higher temperature, the viscosity of the molten salt is lower and leading to lubrication. The corrosion rate increases from 550 to 650 is more pronounced for the 316H. This may be again more corrosion, higher temperatures. The corrosion rate is higher, but it's also maybe a representation. The 316 soften a little bit or more at this temperature and again more prone to wear too.

Looking at the effect of sliding speed, this is a little more complex to understand. Again, as this is mentioning, there's more than one factor here. Not just the sliding speed, but the fact that we maintain the time test constant meant that your sliding distance was much longer as the speed was faster. You have a longer equivalent track over these measurements.

I guess the main takeaway here is that these higher speeds, we also observed that at this higher rotation speed we observe some vibration and we believe that this higher wear volume on the graphite at these hiker speeds may be due to some defect of vibration and the fact that graphite is brittle. So there may be some damage in there due to the vibration at these higher speeds.

Then finally, a comparison of the amount of salt in the test. Just to point out this first graph in here and the last one is basically the original graph that I say going from argon to the presence of salt and these conditions, and also sliding speed – at the lowest sliding speed of 1 millimeter per second again. We go from having a negative wear volume, which again is at the position of graphite on the surface of the stainless steel to having a positive. But we observed that when we have start condition, meaning we don't have a fully submerged interface into the salt, the wear volume and the wear rates increases here. Again, this is because the smaller presence of salt prevented the formation of a self-lubricating graphite transfer film but it was able to provide a stable protective lubricant at that interface.

All of those results were recently put together, and there is a paper that just got published. If anybody's interested in looking at those results in more detail, please look for this publication.

The setup that we have, that we utilized for this, again, was available. It allowed us to do some proof of principle, get some initial data. But we understood that it was the most ideal situation, again, because there's also the possibility that the environment is not completely inert. We have more recently added to our capabilities a new glovebox and tribometer. The tribometer is going to be completely enclosed in our glovebox. This will allow us to handle not only the salt, but the samples and to do the measurements, and again in a complete inert environment. Again, this is just being commissioned. We're doing some testing without closing the glovebox, just to make sure everything is functional. But we're hoping to have this close and getting some additional data pretty soon. We will also be interested in looking at other interfaces. We have, again, some data on graphite on external surface, but we will be interested in looking at graphite on graphite surfaces too.

Just coming to the end of my presentation, just a quick summary. I briefly talked on – the two main topics of interest here were whether salt intrusion into graphite, how we understand that, how we measure that, how we can get information on how that salt is distributed into the graphite sample. This is, again, critical to guide our efforts in trying to understand how that salt is going to affect the properties of graphite, whether mechanical or thermal properties. We also have some efforts here on trying to understand the more fundamental interactions on the salt and graphite by measuring contact angle and potentially developing predictive models of salt intrusion. We have some proof of principle or initial results on where testing of graphite in a molten FLiNaK. We have new facilities that have been installed that would allow us to continue doing this and hopefully expand this to do some erosion testing too.

Next slide is just a summary of the various reports and publications that have been derived, published for the past almost 4 years in this program. The list is here available to you and you guys get the slides. All of these are available at [Unclear] if interested. With that, I would like to thank you and happy to answer any questions that you may have.

Berta
Thank you, Nidia, very much. While questions are coming in, we'll just take a quick look at the upcoming webinars that we have scheduled. In June a panel session on the International Knowledge Management and Preservation of SFR. In July, a presentation on Off-Gas Xenon Detection and Management In Support of MSRs. The July webinar, I'll just give folks a heads up, we are going to test a different platform from our GoTo webinar platform. We're going to test the July webinar on using a Zoom, not Zoom.gov, but Zoom. Further

registration information will be forthcoming on that. In August we'll go back to GoTo Webinar, finish out our subscription that we have with this platform, conclude our webinar series for this year with Corrosion and Cracking of SCWR Materials.

With that, we'll open it up for questions. There are several questions that came in during the presentation. Again, Nidia, if you undock that question pane, it'll help you resize them. But the first one is what graphite are you considering? What MFG, what type? All graphites are not the same as you well know. I believe you addressed that during your presentation. But do you want to talk again about that?

Dr. Nidia Gallego
Correct. I'm trying to see, can you repeat the question again just to make sure that I…

Berta
What graphite are you considering? What MFG, what type?

Dr. Nidia Gallego
I'm not considering any graphite. I'm not building any reactor. I think that's a question that, you know, every reactor designer will have to get hopefully some of the data that we generate in this campaign for a wide range of graphite grades that are available. Salt intrusion is not the only factor that needs to be taken into consideration, so that irradiation behavior, what lifetime, what operating conditions, and so on and so on. All that has to be brought into the equation to be able to decide, I'm going to choose that. But that's not a decision I do. I'm doing this – again, trying to understand more generic and covering a wide range of graphite grades that are available, as you mentioned. As I've shown here, there are different geometries, porosity and grades that are out there.

Berta
Can you please define edge site and basal site?

Dr. Nidia Gallego
Okay, I wanted to say that MFG, and now I understand one, manufacturing, I believe, is what the person is referring to. Again, I cannot mention what are considering or recommending. I cannot recommend a graphite grade. That's just something the designer has to do.

Next question. Basal versus edge sites. Again, just going to that crystal structure, you know, your basal is just your surface. When you have that 2D hexagonal, then you got your plane, that's your basal plane. Your edge site would be the terminating sites for that plane. Again, those are your reactive sites. Those are the sites where you will have activation starts [ph]. Those are the sites that could be reacting with other compounds to [chemical] or have some reaction in there.

Berta
Thank you. Why submerge samples for 12 hours? What drove that timeframe? And did you consider a longer exposure?

Dr. Nidia Gallego
Yes. Our original 12 hours had to do a little bit with our safety in here. That was kind of before we were comfortable leaving the system overnight unattended for longer than 12 hours. That's what we were able to do, like coming early in the morning, having one chief – submerging the sample, and having somebody stay in late and then removing in there. That also kind of is very similar to other values that you see in the literature. You see literature values of 1220 hours. Once we had done this several times and we talked with our [ESH] people, our safety people, and demonstrating that our setup was safe to leave unattended. That's when we went to longer periods of time. Again, that second set of neutron imaging that I showed shows that samples for 12 hours and then 2 weeks. We were able to leave our system immersed on the molten salt for up to 2 weeks.

Berta
Thank you. What are the consequences of salt intrusion on the reactor operation and safety?

Dr. Nidia Gallego
Again, that I don't know. I cannot answer that because it is going to be all dependent on the type of reactor that you have. What are your operating conditions? What are you accounting for? So having the designers, hopefully, once we can provide information, or this amount of salt goes into the graphite grade that you're considering, and it goes into these many millimeters into the surface, it migrates deeper, they can take that into consideration and account that for the operation and account that for the safety case that they may do.

Berta
Thank you. In relation with the publication infiltration of molten fluoride salts and graphite phenomenology and engineering considerations for reactor operations and waste disposal, and cites a journal of nuclear materials, the presentation and the presentation made. To what extent is the impact of the FLiNaK on disposal requirements have been studied theoretically or experimentally, with

or without disposal implementing organizations. That's a long one. I'm going to post.

Dr. Nidia Gallego
Yes, that's a good question, and I really do not have an answer for that. I'm not working on disposal requirements, so I cannot answer that question right now. I'm sorry.

Berta
Thank you. Does irradiated salt intrusion make any difference than non-irradiated salt intrusion?

Dr. Nidia Gallego
I don't know that yet. We are hoping to do some salt intrusions of some irradiated graphite. If the person that is asking this is familiar with irradiation effects on graphite, we know that as you irradiate the neutron dose increases, these dimensional changes causes defects into their potentially – I mean, the porosity may be changing slightly. It is likely that there may be some changes on that, but I have not done measurements. We do have in the plans for next fiscal year to get some irradiated graphite samples and do some comparison to non-irradiated.

Berta
Thank you. Do you have any plans to test other types of fluoride salts?

Dr. Nidia Gallego
So right now we are approved to use FLiNaK. Unfortunately, because of limited budget and all the extreme precautions of handling beryllium containing salt. Right now, at this point, we do not have any plans to move to fluoride. We're doing these more fundamental studies using FLiNaK, but right now, we don't have any plans to move to beryllium containing or other irradiated salts right now.

Berta
Thank you. Could you say a bit more on the time effect? Is there an indication that wetting increases with time, or is there something else that explains changes in the salt penetration with time?

Dr. Nidia Gallego
That is an excellent question, and it's a question that we're asking ourselves and trying to study. Again, we're doing the contact angle, we're looking at different heating profiles, hold times. We see differences. We still cannot explain some of the things that we are observing. Certainly, we're seeing differences. When we do the contact angle measurements. We're doing this flowing argon. So

there's not overpressure to argon. There may be some changes, again, on the salt on how it behaves. What we may be seeing when we do the contact angle may not be 100% representative of what the salt will be seeing when we're doing – when there's an overpressure in there. But certainly, we are trying to understand the time effect, whether there will be other factors, like maybe surface finish or roughness of the surface. Again, certainly the pore structure, that other surface may have an effect on that. So, yes, good question.

Berta
Thank you. How are you considering measuring mechanical or thermal properties of graphite that's been infiltrated with molten salt? Do you have any guidelines to do such measurements?

Dr. Nidia Gallego
That's another excellent question, Mona. I didn't show here, but we did try to do this. Actually, we tried to do this before we did the imaging. I'd always kind of said, okay, do we do it with the salt or without the salt? And this is very challenging because you do these postures, you raise the temperature to have the molten salt, but you got to extract the samples from there. You have to cool down, so it solidifies. Then when you do, if you do your measurements using standard ASTM test methods. Your salt is solid at those temperatures or trying to adapt those testing measurements to high temperatures is not straightforward because, I mean, you not only need to increase the temperature, but you need to make sure you get an inert environment. The question of whether we do the testing with or without the salt is being there. I mean at the ASTM committee, which I participate, we have in that discussion right now. The challenge, though, is removing the salt once it's going into the pores of the graphite. It's not easy, so it doesn't want to come out. What kind of additional damage are you causing to the graphite when you go through that cycle? And as you see, the profile of that salt distribution into the samples is not the same for all. If you were to test with the salt in there, you may have samples where you have just salt accumulated on the edges versus salt across the cross section. That question, again, is being considered under ASTM, but there is no answer yet for that because it's very challenging.

Berta
Thank you. Have you considered how salt intrusion into the graphite and graphite particulars in the salt due to wear affect the characteristics of the radioactive waste that need to be stored and disposed?

Dr. Nidia Gallego

No. Currently, it's not part of the scope of work that I'm addressing, but I do know the community is interested in that. Hopefully, we will have budget available for the appropriate people to be looking into this. We have thought about it, we have talked about it, but not doing any research in that area right now.

Berta
Thank you. Do you expect to conduct similar experiments on impregnated or coated graphite? If yes, what type of impregnations, coatings do you anticipate to explore? Will you explore fuel salts?

Dr. Nidia Gallego
Yes. Right now, I'm not doing any modification to the graphite. I'm utilizing graphite as provided by the manufacturers. I'm not considering any coatings or impregnations of the graphite right now. Again, the question of the fuel salts right now is more an issue of our budget and scope of work still is not there for us to explore the fuel salts. We're doing, again, right now, using FLiNaK most of the work that we're doing.

Berta
Thank you. Based on the testing performed, do you have an impression of the, "optimized graphite?" What are you looking for as favorable parameters resulting from your testing?

Dr. Nidia Gallego
Yeah. Again, this goes back to what I mentioned before. I cannot recommend a graphite, and I believe that what you call an optimized graphite is going to look very different from reactor design to reactor design, based on what operating conditions, what kind of pressures, what kind of temperatures, what kind of flow rates you have, what are you accounting for or not? I don't think there's such a thing as an optimized graphite for a generic molten salt reactor. Again, it's just going to be the reactor designer who will take the information that we're providing, look at what the operating conditions is, deciding what they can live with or what they cannot live with, and how they can make a safety case [the NRC], and then from there, decide the graphite.

I also know there's a lot of other factors besides this, cost of the graphite, shape, size of the graphite available had to be taken into consideration. In my opinion. I don't think there's such a thing as an optimized graphite because there's not just a single reactor design or just a unique operating condition.

Berta

Thank you. I want to take this minute to thank you again, Dr. Gallego, for spending your time with us and sharing your expertise. It's greatly appreciated. That's the questions that have come in thus far. You can tell the interest in the topic. Just given the number of questions, it's always wonderful to see such an engagement. I appreciate your time and willingness to field all of these questions.

Dr. Nidia Gallego
Yeah. And anybody – I know a few of these names in here, so if they would like to reach out, want more information, feel free to contact me. I think my contact information was at the beginning of the slide.

Berta
On the meet the presenter slide is your email. Thank you for that. Thank you for your willingness to go offline and talk with folks more directly. Dr. do you have any concluding thoughts?

Dr. Patricia Paviet
Yes, just thank you so much again, Nidia, thank you. Thank you for the excellent presentation. I wish us to have a little bit more funding for next fiscal year that will help. But thank you. Extremely well presented. Thank you again, Lidia. And again, thank you, Berta.

Berta
You're welcome. Thank you. I'll close it out and we'll let people get back to their day. Thanks so much. Bye-bye.

Dr. Nidia Gallego
Bye. Thank you.

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