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CARBON NANOTUBE FORESTS: THE NEXT STEP IN ENERGY STORAGE?

INTERVIEW WITH DR. WAQAS KHALID

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Dr. Waqas Khalid[1]

***Dr. Waqas Khalid** is a research scholar at the Lawrence Berkeley National Laboratory (LBNL), a project manager at the UC Berkeley College of Engineering, the founder and CEO of the nanotechnology startup company Jadoo Technologies, and a lecturer in the UC Berkeley Department of Physics. In this interview, we discuss Dr. Khalid's work on optimizing and employing carbon nanotube forests for energy storage, biosensing, and beyond.*

BSJ: How did your early experiences growing up in Pakistan shape your scientific career, and what led you to continue those studies in the U.S.?

WK: I was a very smart kid and a big time mama's boy. The goal was to be the best in school, athletics, and extracurricular activities. I did the British education system, the A levels, when I was in Pakistan. After that, I got into a very elite high school. There, the goal was to go to the U.S. for higher education. I was actually asked to apply, but my mom flipped out. She said, "Oh, no, you're not gonna go there. You're never going to come back." My dad was okay with it. It took a whole weekend to convince my mom to allow me to go to the U.S. Eventually she agreed, and I flew here. So my drive for excellence and scientific and academic innovation was always part of my being, so to speak.

BSJ: Why did you go into nanotechnology?

WK: I always wanted to do something really cool and fantastic. I got into a school called Wayne State University in Detroit, Michigan. After the first semester, I got a Global Spartan Scholarship and ended up at Michigan State University. My commute was 200 miles a day, every day, for two semesters. I bought a $1,000 car and I used it to commute, but after two semesters, the car broke down and I ended up having to move back to Wayne State.

While I was at Wayne State, a lot of things happened. Serendipity, I guess. A company called Delphi, which had been a part of General Motors (GM), got kicked out of GM. They spun out as a company of their own, and they built a state-of-the-art cleanroom for micro-fabrication at Wayne State. Then, a new professor from Caltech joined the faculty at Wayne State. He brought his projects from Caltech on making smart skins and biosensors using nanotechnology. I took a MEMS [micro-electromechanical systems] class with him. I ended up working with him for my PhD. I did my undergraduate, master's, and PhD degrees in five and a half years, all three degrees.

BSJ: What are carbon nanotube (CNT) forests, and what are their current applications in nanotechnology?

WK: Graphene is the magic material that everybody knows about. Carbon nanotubes were the predecessors of graphene technology. A sheet of single carbon atoms in a straight format is called graphene. If you roll that sheet into a tube, you will create carbon nanotubes. They have been around for almost 30 years now. They were the promised material before graphene came along, and they have really great electrical properties. They're super good conductors, and depending on how they're folded, they can be semiconductors as well. They can come in an onion-like format, with multiple layers of nanotubes around each other forming multi-walled carbon nanotubes. They can be grown in populations, called "forests," and they also have great optical and thermal properties and a huge surface area.

BSJ: How did you fabricate the CNT forest on chip?

WK: The standard state-of-the-art process is to grow the nanotubes on a surface, peel them from that surface, mix them in a solution, and then use them. I wanted to do an experiment where we grow two pillars of nanostructures, charge one pillar with positive charge and one pillar with negative charge, and electrostatically actuate them. I did some of this work at the University of Michigan, and then I went back to Saudi Arabia to test these devices. They short-circuited through the non-conducting substrate that we had on our devices, the silicon wafer. That was a failed experiment. So from Saudi Arabia, I moved to Chalmers University of Technology in Sweden. There, I built new devices, and then I started a company based on this technology. After four hours of really gruesome discussion with folks at Chalmers Innovation, an incubator for technologies and startups, we realized that nobody in the world has actually ever grown nanostructures that short-circuit through the substrate, and there is no literature about it. I'm the only person who has done it and who can do it. Nobody knew how to grow nanotubes with a good metal contact. The discussion started with questions like, "How do you do it? Why does this happen? What is going on?" I got extremely curious. I thought, "Why don't we do some more experiments and figure out why this happens?" That was the bulk of my work in Sweden. That's where we filed the patents on this innovative idea, detailing how we could build and apply this technology. All great technology is developed by failed experiments and accidents. This is a prime example.

BSJ: What is the purpose of chemically functionalizing CNTs? What are the benefits of functionalizing CNTs on chip, as opposed to conventional methods where CNTs are modified in solution before being redeposited on chip?

WK: When nanotubes grow on a surface, the surface usually contains some metal catalysts. To functionalize carbon nanotubes, people use acids like nitric acid or sulfuric acid to damage the carbon in the nanotubes and make holes. On these holes, -COOH or -OH groups can attach to the nanotubes and functionalize them (Fig. 1). But in this process, the acid actually etches the metal layer and peels the nanotubes from the surface, causing them to float in solution. So this is why most materials that are made of nanotubes are grown on a surface, mixed in a solution, and then redeposited again; it's so they can be functionalized. This was the only way they could be used, technically. While I was in Sweden, this was a huge problem for us as well. At Chalmers University, I worked with Bengt Nordén, who is the chairman of the Nobel Prize committee in Chemistry. He had become a good friend of mine; I met him in Saudi Arabia, and I ended up coming to Sweden to work with him. We came up with a process where we could actually functionalize the nanotubes while they were still on the surface, without peeling them off. We filed a patent on that as well. This protocol now allows you to not only grow the nanotubes in a unique fashion, but also maintain electrical contacts, all in a very cost-effective and simple fabrication process. After growing them on a surface, you can then functionalize them directly on that surface without them peeling off. It allows you to create a very powerful component that can then be used as a powerful device for a lot of different applications in energy storage, biosensing, field emission, or energy harvesting.

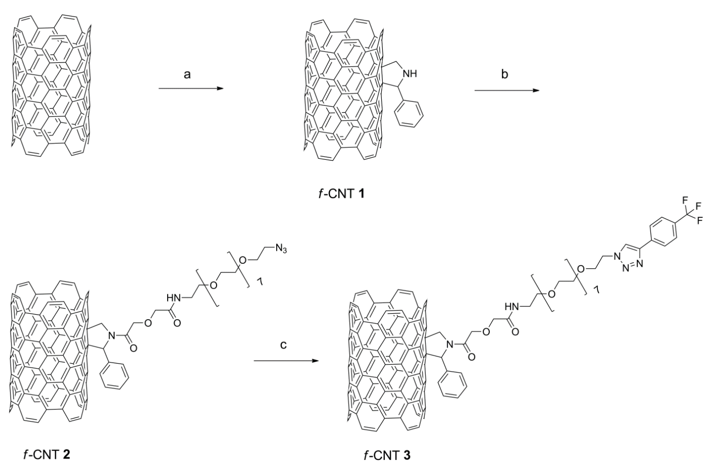

***Figure 1: Carbon nanotubes** functionalized with different chemical groups.*[2]

BSJ: You confirmed functionalization by using scanning electron microscopy (SEM), equipped with energy dispersive X-ray spectroscopy (EDS). Could you briefly explain for readers what SEM and EDS are and how they were used to confirm functionalization?

WK: When you go to the nano- and micro-scale, things are so small that light cannot really give you a good image. So we use electron microscopes. They're pretty big, and they have a vacuum chamber where you suck all the air out and then introduce an electron beam. The electron beam hits the surface of your sample and reflects from the surface in a way that is then picked up by specific detectors. You then have a software that creates an image of the surface. Because electrons are super small, you can get really fine resolution of the surface (Fig. 2).

Once we have an image, the question becomes, “How do we know what materials are on that surface?” To answer this question we can use spectroscopic techniques. We can detect chemicals by the interaction of different waveforms on the surface. Then, different analyzers take the data, and a powerful software can then produce an image or a spectrum to let you know what is going on. EDS does all of that. X-rays fall on the surface, and that interaction with the surface gives you the material properties. If you have a compound with carbon, nitrogen, oxygen, iron, or any other kinds of elements, you will get a spectrum with a peak for each of those particular elements, signifying their presence. This was the technique that we used to confirm that we were actually able to functionalize the nanotubes specifically on a surface.

BSJ: You noted from your SEM data that the technique you employed to functionalize CNT forest on chip preserves the morphology of the CNT forests. What implications does this hold for potential technological applications of your method?

WK: I’ll give you an example. You have a lawn outside. You mow the lawn, take the grass out, and put it on your living room floor. Does that grass have the same morphology and alignment as it did when it was outside in the lawn? Probably not, because grass grows very directionally. It grows vertically, perpendicular to the ground, and it’s nicely organized in a range. But when you chop it off and put it on a surface, the leaves fall flat, and they’re parallel to the surface. That’s what we mean when we say “morphology.” So when you grow nanotubes on a surface, mix them in a solution, and re-deposit them, it’s like the grass from your lawn on your living room floor. In our fabrication process, we can define where to grow these nanostructures—where to grow these grass patches. We provide essential electrical contact with these small patches; it’s like having specific plumbing to water each of these patches individually, wherever they’re grown. Now, this allows you great morphology because you have structures which do not get destroyed and are exactly where you want them to be, since you can define where they are. This fabrication process gives you a platform where you have individually-addressable structures that are very well-defined. Then, you can tap them electrically and chemically to use them in various applications.

BSJ: Continuing our discussion on applications of CNT forest on chip, we wanted to talk to you about your work on energy storage using nanotechnology with Jadoo Technologies. To start, what is the mission of your startup company, Jadoo Technologies?

WK: I started this company a while ago. How do you take a cool concept and build an actual product out of it? Given that I finished school so quickly and developed this technology while I was really young, I was ambitious and enthusiastic. I thought that we could change the world by doing things in a new way. While I was in Sweden, I was offered 15 million Swedish crowns, which is around $3 million, to build this idea and show a working prototype of the technology. But I was of the idea that we could do this in collaboration with academia. Why did we need to burn all this investment capital to get data and lose so much equity early on? We could get the scientific feasibility testing done in a more efficient and effective fashion by grants and collaboration with academia. We could publish papers, distribute the science, and share it with students, rather than having a venture capitalist hold a gun to our head and tell us, “You can only do this, you should not look at any other applications.”

I did a lot of this work while I was in Sweden. I ended up coming to Lawrence Berkeley National Labs to work with Paul Alivisatos, who was the Director of LBNL and is now the Executive Vice Chancellor and Provost at Berkeley. I raised a little grant money here and there and worked with NASA, because NASA wanted to use this technology for monitoring human health in space applications and the human mission to Mars.

While at Berkeley, I started a URAP [Undergraduate Research Apprenticeship Program] project. Since everything was patented, we could use the learning process of rapid prototyping to teach students while getting things done. I became a part of a center called COINS [Center of Integrated Nanomechanical Systems], where a part of my duties was to develop an ecosystem to take technologies developed in an academic environment to an entrepreneurship and industry level.

So, I built the interface for my technology. But to test these things, we had to use these huge commercially available instruments, about the size of a microwave. Okay, you have this very

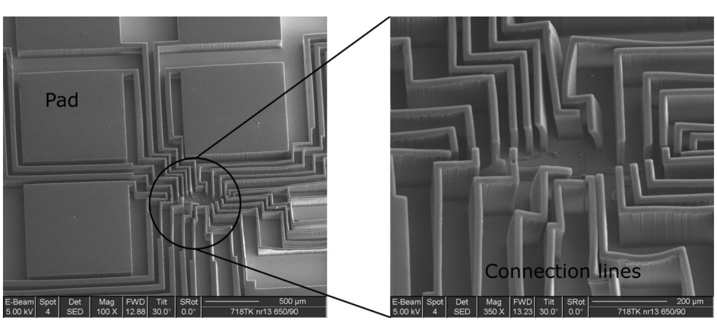

__Figure 2a: SEM micrograph of CNT forest on chip__ prior to functionalization. The CNTs have been grown in a pattern containing pads and connection lines.[2]

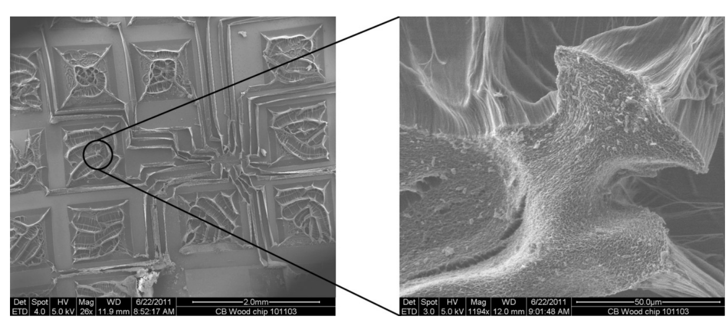

__Figure 2b: SEM micrograph of CNT forest on chip__ after functionalization with an amine group (f-CNT 1 from Fig. 1). Note the preservation of overall morphology: the connection lines to the pads remain well-defined.[2]

small sensing array which can fit on the tip of a hair and has hundreds of sensors. But to use it you need to have something the size of a microwave attached to it, which does not make sense. So, we embarked on the journey of building our own electronics. We also built our own analytical software, using artificial intelligence and machine learning. So now, we have a complete system: the nanotechnology, the interfaces, the electronics, and the software.

This was all done in collaboration with the university, and it's all open source—all this work is shared with researchers at NASA and Berkeley. We are trying to promote the idea that you should share the knowledge, so that rather than everybody doing the same thing over and over again, we can be more efficient. This is one of the many aspects I included when building this new ecosystem, where you could actually mitigate technological and financial risks and create pathways on how to take your product to market. That's the crux of Jadoo Technologies.

BSJ: What are the advantages of using nanocapacitors to store energy as opposed to more conventional modes of energy storage such as lithium ion batteries or supercapacitors?

WK: The idea is the following: energy storage is a huge problem that humans are facing in the modern world. The evolution of clean energy, like solar panels and wind power, are transforming the energy generation landscape. But to store energy, we all use the recently Nobel Prize-awarded technology of lithium-ion batteries.The problems with this technology are that it's slow to charge, it has a limited number of cycles of charging and discharging, and lithium is a limited resource. So, the question becomes: how do you create a new technology platform where you can store charge in a much safer, faster, and cleaner fashion? Once these batteries die out, we'll be left with a huge amount of e-waste that we'll have to clean up again.

I taught a class at Berkeley where students reviewed the different potential applications of our technology. Energy storage was a winner. Our technology can provide a very unique storage solution. You can create these capacitors, called nanocapacitors, and you can put a lot of them together on a smaller chip. That will give you a capacitance unlike anything else previously built. We were very enthusiastic, performing a lot of calculations and simulations to show that our technology could actually work. Then, I gained access to Lawrence Berkeley National Labs through a proposal process, where I built a few prototypes and showed that the technology really works. We also got some preliminary data, which looks extremely promising. On the size of a postage stamp, we can store enough charge to power toys and electronics, more than a 9V battery. So, if successful, we could actually create an energy storage solution that could charge instantly. You can create wireless energy charging protocols with this technology. The problem is, many people think our technology is too good to be true. Everybody wants to see a working prototype and more data. Who knows what will happen? The best thing is to show a working prototype.

There are also other interesting electric materials coming out in the market called colossal dielectric materials. These materials don't really exist in nature. What we do is put atomic layers of these materials on our nanostructures and build different materials on top of each other, creating a new kind of material that has really tremendous energy storage properties. Combinations of these cutting-edge technologies allows us to build an energy storage solution that can revolutionize the future of energy storage if successful.

BSJ: Could you tell us about your journey as an entrepreneur? What unique challenges have you faced along the way?

WK: Life has been full of interesting twists and turns. Once I finished my PhD, I moved to California and started working at a company called Brocade Communications. I went to Mexico to get my H-1 visa situation sorted out. Somebody at the U.S. border detained me and put me in detention for 37 days. They deported me to Pakistan and gave me a five-year ban from the U.S. I was lucky enough that I had a visa for Canada. I ended up in Canada. The U.S. government later said somebody made a mistake, and they fixed the situation and removed my ban. I lost my job during this process, but while I was in Canada, I started working with the University of British Columbia and got exposed to carbon nanotubes. Bringing in my knowledge of MEMS, we developed some biosensors and got some really cool data. This led to me moving to KAUST [King Abdullah University of Science and Technology] in Saudi Arabia and continuing my entrepreneurial endeavors, trying to see how we could build this new idea that I had. These events led to me interacting with Bengt Nordén, to my work in Sweden building up my IP portfolio, coming here to Berkeley and LBNL, meeting NASA folks; life just took its own turns.

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