Processing ceramic composites using microwaves and RF at the University of Birmingham

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I have been invited to write a short article about the research being done in my research team that involves the use of microwave and radio frequency energy. Those who know me will know that this has to be associated with the processing of advanced ceramics and composites; materials with which I have been associated for over 45 years.

Historically, my team has investigated the potential for use the microwaves in a wide range of ceramic process routes – and for a very diverse range of ceramics. The processes include (but are not limited to) sintering, joining, slip casting, binder burn out and synthesis. To back this up we also devoted much time to exploring the so-called 'microwave effect' back in the 1990s and to dielectric measurements. Famously, all this work led to the publication of a deliberately controversial paper entitled *When Should Microwaves Be Used In the Processing of Ceramics* [1]. I remember presenting the paper at a conference at about the same time and had the misfortune to precede a speaker who went on to present their work on a topic that I had firmly put in the 'where microwaves should not be used category'; even more unfortunately, their results absolutely supported my hypothesis!

Since those early days we have focused on investigating those routes where I believed – and still believe – that there is a clear advantage to be had from using microwave, or RF, energy. The number one (but not the only) area has therefore been a process known as chemical vapour infiltration, CVI. In this process a gas is heated as it flows through a porous body to a temperature where it breaks down to yield a solid. As such, the process is closely related to the better-known chemical vapour deposition, CVD, which is used to produce a wide range of coatings from different gases. In CVI, in which the reactions are the same, the gas deposits a matrix inside the porous structure – the best known example is the production of commercial aircraft brakes. Methane is the gas and it flows through woven carbon fibre structures, known as preforms, that are heated to about 1000°C. The methane breaks down to yield carbon, creating carbon fibre / carbon matrix (or carbon / carbon, C / C) composites.

Although I have worked on these composites before – back in the 1990s I helped Dunlop Aviation to create a faster process for making aircraft brakes that I believe was commercialised – our work these days focuses on two main types of composites. The first are essentially carbon / carbon composites but which also contain specific ceramic powders, known as ultra-high temperature ceramics (UHTCs) [2].

Examples include quite specialised materials including zirconium diboride, ZrB_2, and hafnium diboride, HfB_2. The former composites have been demonstrated to survive temperatures of around 2500°C *combined simultaneously* with Mach 4 – 5 gas flows, whilst the latter can manage around 3000°C, again combined with hypersonic gas flows [3]. It has also been shown that our materials can survive multiple exposures to these conditions, so they exhibit reusability [4]. It won't be a surprise, therefore, to learn that these materials are being seriously considered for two main applications; hypersonics, e.g. leading edges for hypersonic craft, and the inner lining for rocket nozzles (though the latter are unlikely to need reusability!). So far, our materials have passed just about every test that has been thrown at them by potential end-users [5].

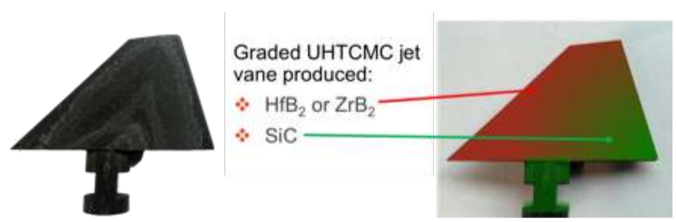
The second type of composites are made of silicon carbide, SiC, fibres infiltrated with a silicon carbide matrix; the so-called SiC / SiC composites [6]. These are now starting to be used fairly extensively in a range of aerospace applications since they can survive around 1700ºC and are much lighter than titanium- and nickel-based alloys, providing more efficient jet engines amongst many other applications.

So, what is the role of microwaves and RF? The biggest issue for the CVI process is that it is SLOW. It can take around 1000 hours to make an aircraft brake – albeit they are typically made in huge chambers that can hold up to around 1000 brakes. The problem is that the initial woven fibre preforms need heating very slowly indeed, typically at something like 1ºC min^{-1}. This is to minimise temperature gradients. Unfortunately, they still do get created and so deposition can occur preferentially in the surface layers and on the outside of the preform. This blocks the flow of the gas through the preform, slowing the process down further. The solution is to cool the system, remove all the parts, machine their surfaces to reopen the required channels, then reheat them back to the process temperature and start again. It can take 2 or even 3 of these steps before the parts are finished – in total, a period of 2 to 3 months.

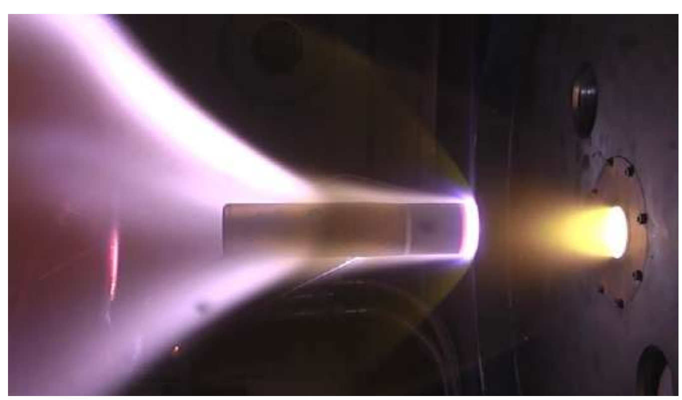
Many research teams have investigated approaches to speed the process up, including temperature gradients, pressure gradients and forced flow of the gases – including reversing the gas flow periodically. To the best of my knowledge, none have been commercialised. Our approach is to use microwaves when heating silicon carbide fibres and RF when heating carbon fibres; the two types of fibres couple particularly well with the appropriate frequency.

This means that now heating is from the 'inside-out'. What is known as an inverse temperature profile develops, i.e. the inside is hotter than the surface. Deposition therefore occurs first in or near the centre of the preform and then, as it builds up, the gases are increasingly forced through the surface layers allowing the whole preform to be infiltrated with the desired matrix [7]. For reasons that, irritatingly!, I don't know, the use of microwaves can enhance deposition times for SiC / SiC composites by about an order of magnitude, whilst RF can speed up the creation of C / UHTC composites by up to a factor of about 40. We can therefore make our hypersonic / rocket nozzles composites in as little as about 24 hours. Naturally, this is really rather beneficial for the economics of the process; whilst they are generally good when making thousands of aircraft brakes, they are less good when the component is only needed in the hundreds or less. Speeding up the process is the 'holy grail' for making components in relatively small batches.

The University of Pisa in Italy is doing some super work on scaling up the microwave CVI route to SiC / SiC composites, whilst we are focusing more on the C / UHTC composites. We can now make composites measuring up to around 30 cm in diameter and several centimetres thick – and can also produce 'graded' composites, in which the composition varies throughout the component. This allow us, for example, to put the most expensive (and dense) ceramic at the surface where it is needed and a cheaper (and less dense) ceramic further inside the component with a gradual change in composition between the two, avoiding stress build up from a sharp interface with different thermal expansions on either side.



Hypersonic jet vane and an example of the type of grading that is now possible.



The carbon fibre / UHTC composites being tested at 2500ºC in an arc-jet.

At this point I would like to introduce you to two of my research team who have done the most recent work on the C / UHTC composites; Drs Vinothini (Vinu) Venkatachalam and Rebecca (Becky) Steadman since I cannot pretend that I have done it all myself. Vinu is actually a former PhD student of mine from my Loughborough University days and, after several years in industry, she decided she couldn't live without me … – seriously, I was utterly delighted when she applied to join my group again a few years ago. Following on from a previous, and also super, postdoc, Dr Virtudes Rubio, she has worked on a range of contracts from Europe, the UK and the military. Without her the scaling up would not have happened. Becky, who is also a former PhD student but who moved straight into the role of postdoc at the end of her higher degree just a couple of years ago, has very much focused on the production of graded materials. Between them, they are a real force to be reckoned with! I have been so lucky with so many of my research team; talented individuals who are hard working and real team players – who could ask for more?!

So, where next? Much of my teams' current work is focused on ceramic composites, both the types described above as well as other types and those made by other routes. Key goals are to produce what I call a 'buffet table' of composites – with a wide range of tuneable properties that can be easily manipulated to suit whatever new application comes along – and to reduce the cost of the composites since this is a major factor holding back their exploitation. Amongst other things, we are working on oxide ceramic-based composites, also for aerospace applications, additive manufacturing of ceramic composites, joining of ceramics and composites to each other and to metals, and are currently reviewing the potential for ceramic composites in nuclear fusion. It is certainly keeping us busy!

For further reading

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About the author

Prof. Jon Binner is the Chair of Ceramic Science & Engineering in the School of Metallurgy and Materials, at the University of Birmingham. He has published over 260 research papers, as well as editing or contributing to 24 books, given around 90 keynote, plenary and invited talks at international conferences and holds 7 patents. He is a Fellow of Ampere, the European Ceramic Society (ECerS), the American Ceramic Society (ACerS) and the Institute of Materials, Minerals & Mining (IOM3). He was the President of ECerS from 2019-21, the President of the IOM3 from 2012-14 and has been the UK's representative on the International Ceramics Federation since 2011. He has supervised over 50 PhD students and even more postdocs and has won numerous prizes during his career. He is very proud of the fact that, despite working in an engineering subject, since the start of his career ~40% of his research team have been female and ~50% of them have been from ethnic minorities. He is constantly looking to improve the fraction that is female.