

## Whiskers at Waltham Abbey

Ann Clarke's mention of whiskers in the winter 2018 issue of Touchpaper stirred some very distant memories for me. In 1956 I joined a Tube Investments materials research group, headed by James Gordon, on the lookout for novel structural materials when reports of very fine metallic filaments (promptly called "whiskers") growing spontaneously on plated metal surfaces began appearing in scientific journals. (I discovered that there were some on the drawer runners of my filing cabinet.) Numerous reports followed and it was suspected that the fibres were unusually strong for their size. A colleague duly designed and made a miniature tensile testing instrument which confirmed that many had strengths close to theoretical estimates.

Jim Gordon, who had previously worked at RAE Farnborough with glass and asbestos fibres, wondered if non metallic materials generally could also be made in strong whisker form to make a new generation of fibre reinforced plastics to replace existing glass-resin composites. Our research group set about studying a range of everyday materials and we concluded that if you could find exactly the right crystallising conditions most could be made in filamentary form. Top material on Gordon's wish list was the refractory silicon carbide known to have a very high Young's Modulus (stiffness), ideal as a reinforcing fibre. This was quite a challenge to make needing near white heat which stretched our lab furnace beyond its limits and to an early grave, but in the end we had a tiny sample of silicon carbide whiskers. We had to think bigger.

The result was a gas tight cylindrical drum, about the size of a dustbin, lined with insulation and heated by a central carbon electrode. It was promptly named the "Bran Tub" as any outcome was a lucky dip. Early experiments were encouraging. We managed to grow a few grams or so of tiny straight filaments of silicon carbide on a carbon substrate. Beginners luck.

It was just at this point that we, as a research group, were given our marching orders by our employer Tube Investments which was suffering financially as a result of abruptly changed company tax laws. Our work was not considered to be relevant to the manufacture of steel tubes. Thanks to Jim Gordon with contacts in the Ministry of Aviation we were offered premises at ERDE Waltham Abbey when Dr. Johnson was the director. (He was a gentleman of the 'Old School' who used to walk his dog in Powder Mill lane in order to wish late comers a curt 'Good Morning'!) He made us very welcome.

Our move in 1962 to comfortable premises on L157 North Site took six months where Jim Gordon was installed as Superintendent of a new Materials 2 Branch bringing with him Noel Parratt, Ron Gooding, Mike Dyne and myself. Long term resident Tom Lewis was co-opted to assist us Materials freaks with basic chemistry. Once settled in, we broached the possibility of making a scaled up version of our Bran Tub at a meeting with the ERDE Engineering Branch. As we feared this got a pretty startled reception from a team used to doing nearly everything in an explosives environment with steam and compressed air whereas we were talking about very high temperatures and a massive electricity load. Eventually it was agreed that we should liaise with the Design Office to sketch out our requirements. This was the beginning of a long exchange with Allen Clarke who relished the novel challenge. Overall, about 100 KW of heating power might be needed, necessitating water cooling for the reactor case. Building L168 looked the ideal site, currently holding the remains of a beam engine. It already had high level water tanks suitable for cooling and was close to the substation.

Fast forward five years of plans, building modifications, specifications for tender, assorted contractors, etc. and we had two giant Bran Tubs ready to be commissioned (two tubs so that they could be run in tandem, one heating whilst the other cooled). Mike Dyne had drawn the short straw to stay the night on the first trial. All went well and the solid state temperature control  $\pm 3^\circ$  at  $1450^\circ\text{C}$  was probably a first in 1967, thanks to a very enlightened electrical

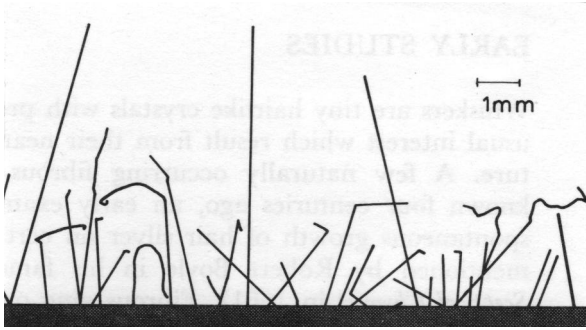
contractor. We then tried a series of silicon carbide runs only to find small patches of whiskers scattered around the walls much as in our earlier small scale trials. With the prospect of the 1968 Open Day looming we hastily resorted to a successful silicon nitride whisker run which looked like freshly fallen snow. That, probably, is what Ann remembers.

Subsequently, with Tom Lewis's direction we tried other unsuccessful routes to silicon carbide, one involving corrosive silicon tetrachloride with a hydrogen atmosphere. By now we were fairly sure that, in spite of the carbon box lining the heated reaction zone, much of the problem was coming from some volatile component emerging from the brick insulation which persisted from run to run. At the time there were no alternative high temperature bricks available. (Our experiments in the original small Bran Tub had used expensive carbon fibre insulation). About this time the Americans published a paper showing that silicon whiskers could be 'seeded' to order in sterile conditions using radio frequency heating. Perfect for the scale of semiconductor manufacture but not a feasible approach for us. Meanwhile RAE Farnborough had succeeded in converting commercially available polyacrylonitrile fibres into moderately strong pure carbon fibres.

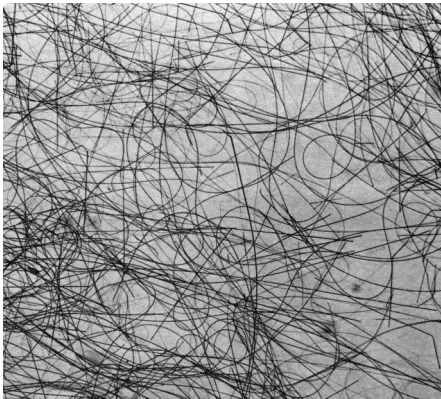
The carbon fibre composite era had arrived.

## **Chris Evans**

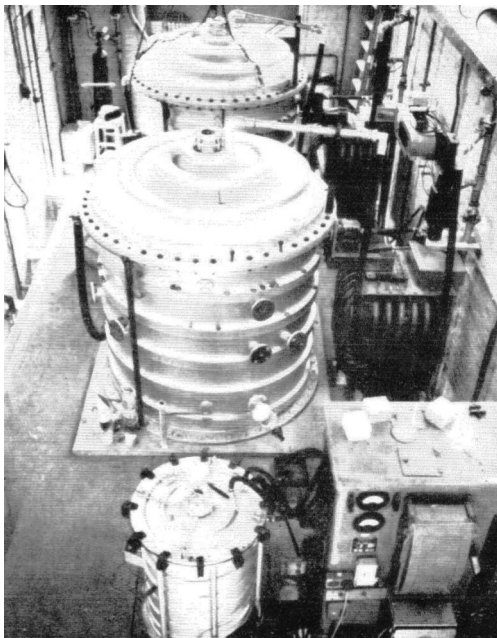
*James Edward Gordon (1913 - 1998) had the gift of writing about technical matters in an entertaining and readable style resulting in two best selling Pelican Books on Strong Materials and on Structures which were translated into at least twenty languages for use as course textbooks. He moved on to accept the newly created Chair of Materials Science at Reading University where he made many more contacts whilst enjoying the academic life. Cambridge University named a laboratory in his memory. In his younger days he was a keen yachtsman and crewed in Atlantic Ocean races.*



**Tin Whiskers**



**Silicon Carbide Whiskers**



**Bran Tubs**