LINCET Project - Minutes of 2nd Progress Meeting

Mat. Sci. Dept., Cambridge: 20th September 2016

Attending

Bill Clyne twc10@cam.ac.uk	TWC	Cambridge U. (UCAM)
Olivier Guillon o.guillon@fz-juelich.de	OG	Jülich (FZJ)
Robert Vassen r.vassen@fz-juelich.de	RV	Jülich (FZJ)
Dietmar Koch Dietmar.Koch@dlr.de	DK	DLR Stuttgart (DLR-S)
Paul Mantle pam73@cam.ac.uk	PAM	Cambridge U. (UCAM)

For Information

Roger Reed roger.reed@eng.ox.ac.uk	RCR	Oxford U. (UOX)
Robert Singer robert.singer@ww.uni-erlangen.de	RJS	Erlangen U. (FAU)
Hans-Peter Bossmann Hans-peter.bossmann@ansaldoenergia.com	HPB	Ansaldo Energia (AE)
Lee Marston Lee.Marston@dynamic-materials.com	LM	Fiberstone (FS)
James Dean jd362@cam.ac.uk	JD	Cambridge U. (UCAM)

2.1 Welcome and Apologies for Absence

TWC welcomed everyone and expressed his thanks for their attendance. There were no apologies.

2.2 Establishment of the LINCET grant and brief financial update

The project has now been running for about 5 months. The main activities during that period have been a DoITPoMS summer school and some preliminary experimental work involving HIPing of Fiberstone. PAM provided a brief financial summary and presented the table below. Future summaries will include comparisons between projected and actual rates of spend, but for the present it should just be noted that the expenditure to date is approximately as planned.

Heading	Budget	Expenditure to date (May – Sept. 2016)	Balance
Staff	£52,912	£11,989	£40,923
Travel & Subsistence	£18,000	£457	£17,543
Other	£52,840	£1,570	£51,270
Total	£123,752	£14,016	£109,736

2.3 The LINCET website - see http://www.ccg.msm.cam.ac.uk/initiatives/lincet

PAM confirmed that the website is now active and contains some basic information about the project. Further material will be uploaded as appropriate, and PAM made it clear that contributions are welcome from everyone involved in the project. In particular, there is a plan to provide information about equipment that could be used during the project. At present, there are no restricted access parts of the site.

2.4 Outcome of the DoITPoMS summer school

The DoITPoMS summer schools are focussed on creation and updating of web-based Materials Science teaching and research resources (www.doitpoms.ac.uk/), with Cambridge undergraduates employed in the Department. This took place, as planned, from 4th July – 26th August this year. Three new Teaching and Learning Packages (TLPs) were created, all in areas of relevance to the LINCET project. The following three students and supervisors were involved:

- Luke Diana - "*Tribology and Wear*" (Kevin Knowles)
- Radu Bizga - "*The Finite Element Method*" (James Dean)
- Arthur Keunzi - "*Powder Processing*" (Bill Clyne)

A report on the summer school is attached as Appendix I.

2.5 Outcome of the first trial on HIPing of Fiberstone

Initial work has been focused on attempts to reduce the porosity level of Fiberstone (www.fiberstone.co.uk/) material, using Hot Isostatic Pressing (HIP). A brief report on the outcome is attached, as Appendix II. The HIP process did reduce the porosity level considerably, although the particular samples used for this work contained exceptionally high levels (~40-50%), probably because they were "cast" into non-absorbent moulds. Levels were reduced by the HIPing to ~5-10%, but this is still too high to expect much improvement in (metal fibre) oxidation resistance. (Virtually all of the porosity, initially and after HIPing, is surface-connected.) Further work, using material with lower initial porosity levels, is planned.

2.6 Future experimental activities, visits etc

It was agreed that there is considerable scope for further work following up on the preliminary activities, and this need not be limited to studies of Fiberstone, provided it is related to materials (and coatings) for mechanically-demanding applications at high temperature. Short visits (one or two weeks) or slightly longer ones (one or two months) are considered to be most promising, probably for the most part by German students / researchers to Cambridge, with the summer period (early July to late September) being organisationally most convenient. This will be explored further over the next few months.

2.7 Arrangements for the first LINCET Symposium (Downing, 30/31-3-17)

A booking has been made at Downing College, for the first LINCET Symposium, for 30th/31st March 2017. This is a Thursday/Friday, and a similar format to HELSMAC will be used. However, the meeting will be smaller and more focussed, with a planned attendance of about 20 (the majority of whom will be expected to give brief talks). The budget of the project includes a sum of £12.5k in support of this Symposium. This should cover the costs in Downing (lecture theatre hire, accommodation for all delegates and all meals etc), plus travel costs for all delegates (up to a maximum, which will probably be about £250). Progress was made on drawing up the list of invitees and TWC will circulate the invitations within a week or two.

2.8 Date of next meeting (at first LINCET Symposium)

It was agreed that the next meeting will be held in Cambridge during the 1st LINCET Symposium. The members of the Advisory Board will be invited to attend.

2.9 Any Other Business

There was none.

Appendix I: DoITPoMS Summer School 2016

The summer school ran for 8 weeks, from 4th July until 26th August, involving three students, one professional programmer (David Brook) and three academic consultants (plus some other members of staff assisting, particularly Lianne Sallows, who acts as the Department Webmaster). The funding was provided entirely from the LINCET project. The following Teaching and Learning Packages (TLPs) were created:

- "*Powder Processing*"
  see http://www.doitpoms.ac.uk/tlplib/powder/index.php
- "*The Finite Element Method* "
  see http://www.doitpoms.ac.uk/tlplib/FEM/index.php
- "*Tribology and Wear* "
  see http://www.doitpoms.ac.uk/tlplib/tribology/index.php

These have all been reviewed and approved, and are now on the public DoITPoMS site. Of these TLPs, Powder Processing is probably the one that is most relevant to the LINCET project, which is partly aimed at improving the (powder-based) processing of Fiberstone-like material. Of course, these packages are teaching aids, and don't have advanced research-oriented coverage, but they do include interactive resources that can provide useful scientific background to research. That TLP does include a brief treatment of HIPing, as well as conventional pressing and sintering - see www.doitpoms.ac.uk/tlplib/powder/consolidation.php.

Appendix II: First Trial on Hot Isostatic Pressing of Fiberstone

Collaborative Partners: Fiberstone, FZ Jülich, DLR Stuttgart

Introduction and Summary

The main objective is to establish whether a processing route can be devised that leads to a Fiberstone-like material that is essentially free of porosity. It should perhaps be emphasized, not just that porosity levels are high in conventional Fiberstone, but that, for many applications, this is not a significant problem. Fiberstone is a very tough material, with the toughness almost entirely conferred by the presence of the (coarse) metallic fibres [1]. Porosity in the matrix does not affect the toughness. However, it does allow the atmosphere (normally air) to enter the matrix and oxidize the fibres, which does slowly degrade the mechanical properties of the material, depending on the oxidation resistance of the grade of fibres being used [2]. There is therefore an incentive to eliminate the (surface-connected) porosity in Fiberstone, for usage in highly demanding applications.

The plans for a summer placement student and an EPSRC CASE PhD studentship did not come to fruition, so the human resources available for this work have been limited over the past few months, and this will continue to be the case over the coming months. Nevertheless, a short exploratory investigation has been undertaken, with some composite cylinders being produced at Fiberstone and transferred to FZJ for canning and HIPing. These were then returned to Cambridge, where they have been investigated microstructurally (mainly via X-ray Computed Tomography). The cooperation of Lee Marston, Robert Vassen, Ralf Steinert and James Dean in carrying out these investigations is much appreciated.

There has thus been only a very limited investigation so far, and it has been focused exclusively on the pore level and architecture in the material. Also, it has involved only one HIP experiment and one variant of the Fiberstone material. Details are provided below, but the broad outcome is that the porosity level, which was very high (~40%) in the particular Fiberstone samples that were provided, was sharply reduced (to ~5-10%). This is obviously encouraging, and suggests that significant deformation of the ceramic matrix took place under the HIP conditions - ie at a temperature of 1200˚C. However, the residual porosity level was still relatively high. Also, while much of it was uniformly dispersed in the matrix, there were some large voids. It seems likely that it was difficult for the can to accommodate a macroscopic shape and volume change sufficient to remove all of the porosity. The sample diameter was reduced during HIPing from 38 mm to 36 mm (representing ~15% volume reduction, if the axial strain was the same as the transverse strain). There will certainly be a limit to the (uniform) shape change that can be tolerated by the can and it was noted that intrusions formed at one or two places on the surface of the can. It is possible that, by starting with less porous Fiberstone (and material can certainly be produced with no more than 20% porosity), a HIP operation could result in reduction of (surface-connected) porosity to a suitably low level (<~1%). No investigations have been instigated so far regarding the effect of HIPing on oxidation resistance, mechanical properties etc.

Experimental Procedures

Production of Fiberstone Samples

Fiberstone is normally "cast" into wooden moulds. This is convenient for a material that is frequently produced in complex shapes on short production runs, but it also seems likely that using moulds with a capacity to absorb water is beneficial in reducing the porosity level of the product. (Much of the porosity in Fiberstone is thought to originate from excess water becoming trapped in the material after the hydration reactions are complete: usage of lower water levels in the slurry means that its fluidity is insufficient to infiltrate fully into the assembly of metallic fibres.)

For the current experiments, however, the slurry was poured into steel moulds, which were to serve as the cans for the HIP operation. This appeared logical and convenient, but appeared to result in porosity levels that were higher than normal. Excess water is routinely eliminated via a post-casting heat treatment at relatively low temperature (~400˚C), but this does not remove the pores in which the water had accummulated. These Fiberstone samples contained about 10-12vol% of AISI 304 stainless steel fibres, which were produced by melt spinning. Fibre diameters were about 0.5 mm, and lengths about 20 mm.

HIPing

HIPing was carried out at FZJ, using stainless steel cans of internal diameter about 38 mm. The cans were evacuated prior to sealing off. The HIPing process was carried out at 1200˚C and 200 MPa (2000 bar), for a period of 2 hours. The sample was then cooled and the can was machined off.

X-Ray Computed Tomography

The X-ray radiographs were obtained using a Bruker Skyscan 1172. The resolution of these scans was about 2 µm (so each 3-D voxel was a cube of side 2 µm). This allows capture of the main characteristics of the pore architecture in Fiberstone: fine porosity has been investigated using mercury porosimetry - see below. Samples were cut from both the original Fiberstone and the HIPed material. While sample sizes were typically several mm in linear dimensions, using this resolution would lead to files too large for tractable meshing if they covered the whole sample. Sub-domains were therefore used, typically with sides of the order of 100 µm in length.

Mercury Intrusion Porosimetry (MIP)

MIP has been carried out using a MicroMeritics AutoPore IV (Norcross, GA, USA). This is mainly oriented towards fine porosity (since the pressure needed to infiltrate coarse pores tends to be too low to measure accurately). The Fiberstone matrix does contain fine porosity (below the resolution of the XR-CT) in the "solid" material surrounding the coarse pores. Its nature is illustrated by Fig.1, which shows an MIP pore size distribution and a high magnification SEM micrograph of such a "solid" region, together with a lower magnification micrograph in which larger pores can be seen. There is clearly sub-micron porosity present, although it normally constitutes only a small proportion of the overall porosity – perhaps ~2%, compared with ~20-30% for the coarse porosity. It's thought that at least most of the coarse porosity was occupied by (excess) water while setting took place, while the fine porosity arose during the hydration reactions, which generate a lot of very fine particulate.

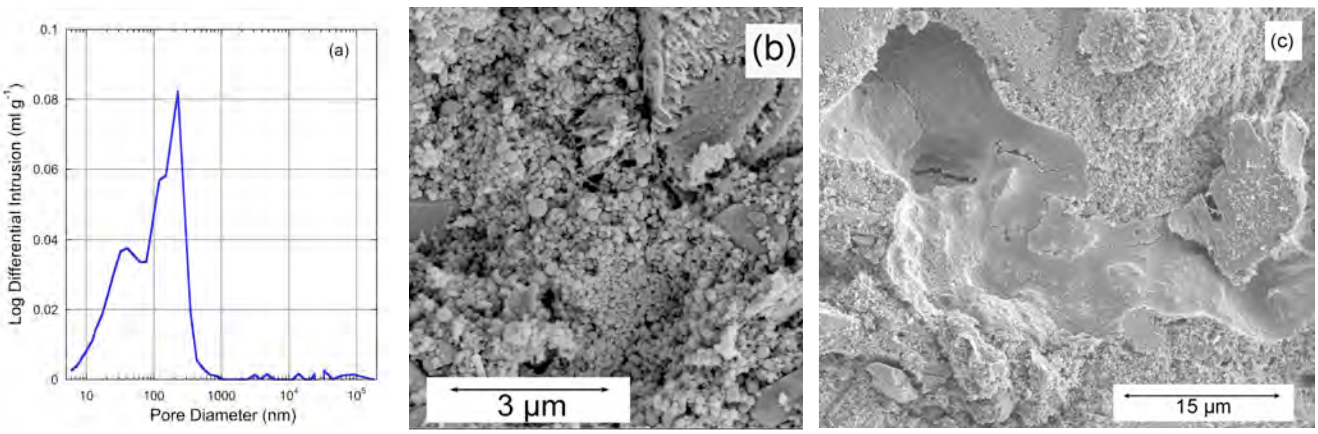

Fig.1 Fine scale pore architecture in a typical Fiberstone sample, illustrated by (a) an MIP (fine scale) pore size distribution, and SEM micrographs of fracture surfaces at (b) high and (c) low magnifications.

Tomography Results

Tomographic reconstructions from an unHIPed sample are shown in Figs.2 and 3. This sample contained no fibres. The reconstruction shows the porosity (in blue) - the surrounding ceramic has been removed. It can be seen that, in addition to the large void (diameter ~ 500 µm), there is extensive dispersed porosity that is quite coarse (diameter ~ 10 µm), with a very high overall level. (The figure of 49% for the small region shown is a little higher than the average, which was of the order of 40%, but obviously these are very high levels.)

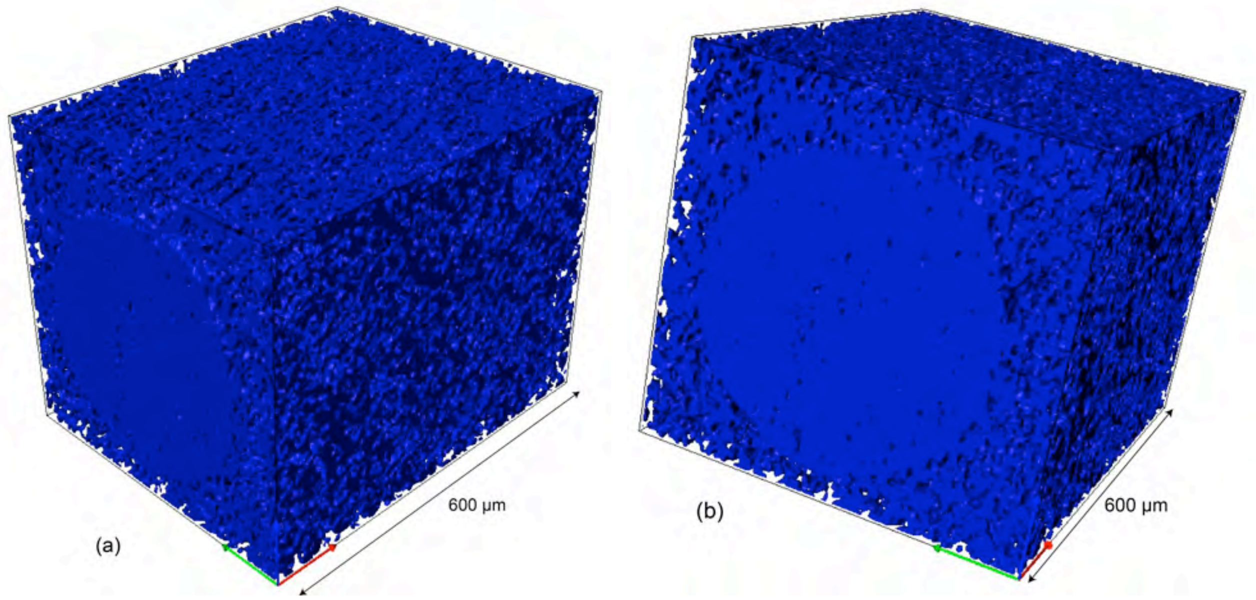

Fig.2 Perspective views, from two viewpoints, of a tomographic reconstruction of a region of the matrix of the untreated Fiberstone material. The porosity is coloured blue and it can be seen that this sample contains one large void, in addition to extensive dispersed (interconnected) porosity.

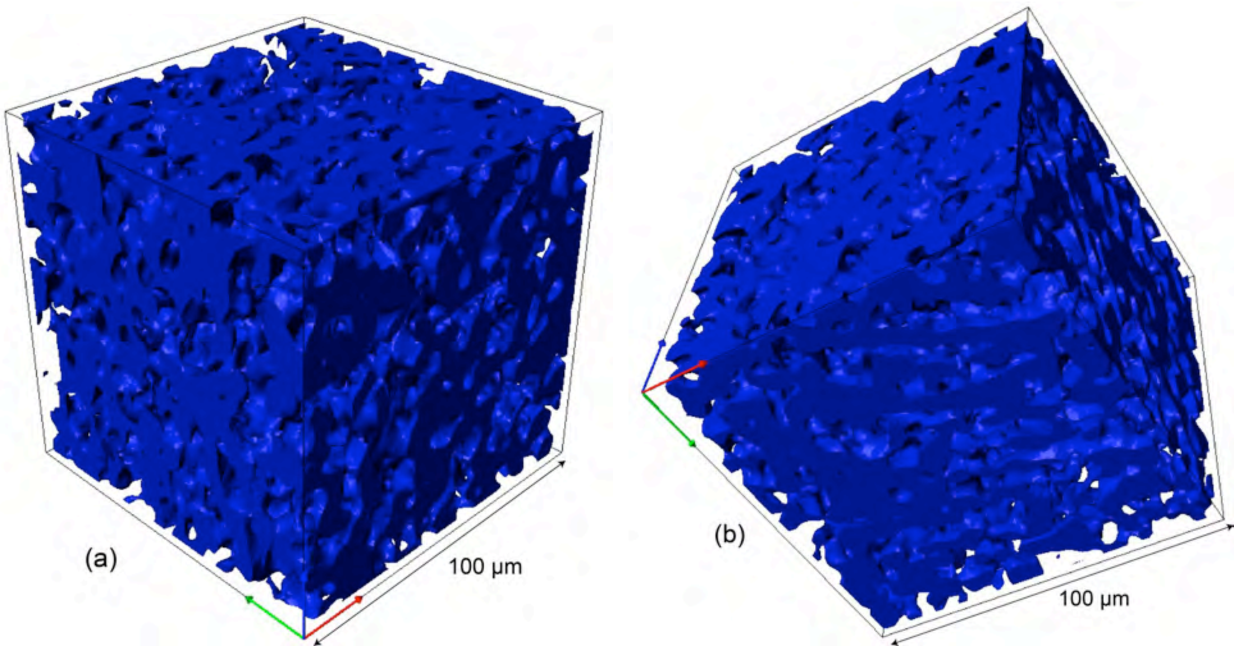

Fig.3 Perspective views, from two viewpoints, of a tomographic reconstruction of a small part of the region shown in Fig.2. The porosity level in this volume is about 49%.

After the HIPing operation, these levels had been substantially reduced. Fig.4 shows a tomograph from a large region of the HIPed sample, which contained sections of several fibres (shown in red). Only a few pores in this volume were relatively large (>~30 µm), although the complete HIPed sample did contain a few very large voids. Also, it can be seen that there were no large pores close to the fibres. On examining a typical region of the matrix more closely (Fig.5), it can be seen that there is in fact some dispersed porosity throughout, although the level is much lower than was typical of the unHIPed sample.

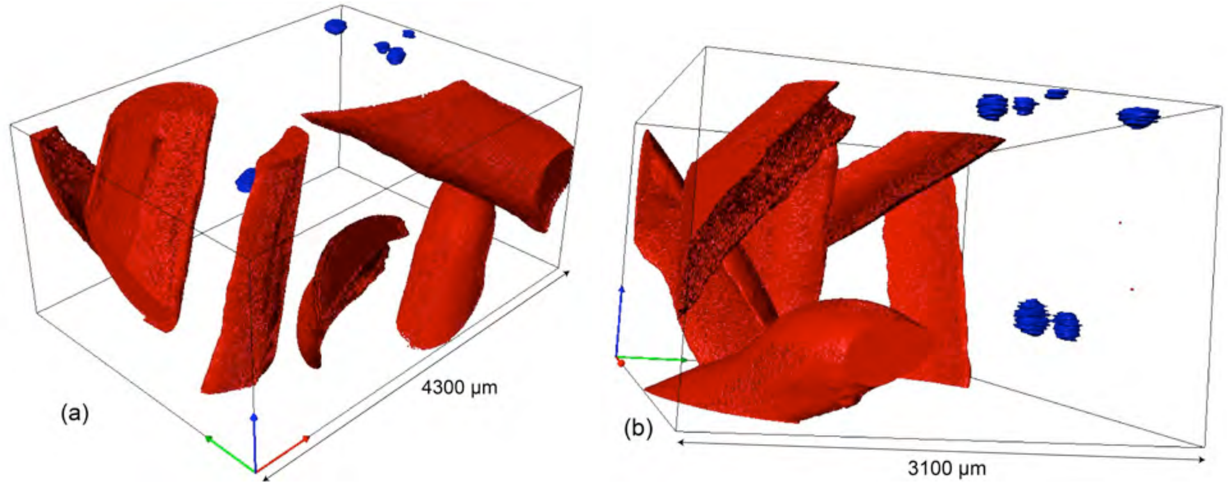

Fig.4 Perspective views, from two viewpoints, of a tomographic reconstruction of a region of the HIPed Fiberstone material. The porosity is coloured blue and the fibres are coloured red. Only the larger pores in this sample are shown here.

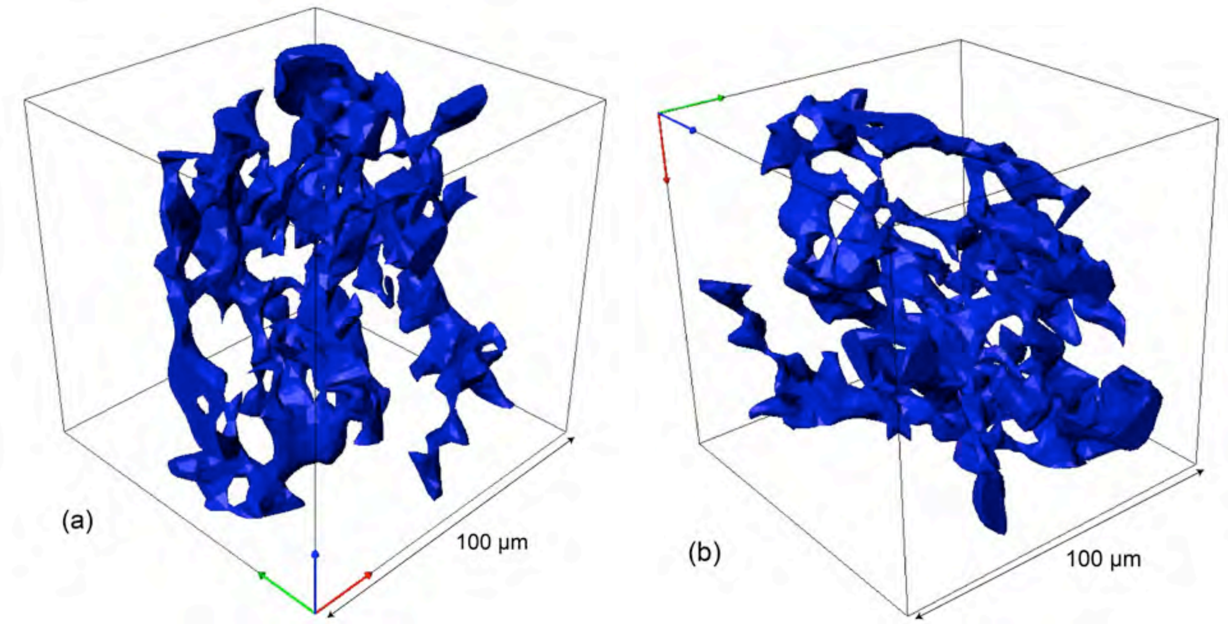

Fig.5 Perspective views, from two viewpoints, of a tomographic reconstruction of a small part of the (matrix) region in Fig.4. The porosity level in this volume is about 7.5%.

References

[1] SR Pemberton, EK Oberg, J Dean, D Tsarouchas, AE Markaki, L Marston & TW Clyne, *The Fracture Energy of Metal Fibre Reinforced Ceramic Composites (MFCs)*, Comp. Sci. & Techn, **71** (2009) p.266-275. doi:10.1016/j.compscitech.2010.10.011

[2] SK Lam & TW Clyne, *Toughness of Metal Fibre / Ceramic Matrix Composites (MFCs) after Severe Heat Treatments*, Mat. Sci. & Techn. **30** (2013) p.1135-41. doi:10.1179/1743284713Y.0000000413