STRP 031712

INTERFACE

Interfacial Engineering in Copper Carbon Nanofibre Composites (Cu-C MMCs) for high thermally loaded applications

SPECIFIC TARGETED RESEARCH PROJECT

SIXTH FRAMEWORK PROGRAMME

PRIORITY 3
Nanotechnologies and nano-sciences, knowledge-based multifunctional materials and new production processes and devices

Publishable Final Activity Report

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1 Project Execution

1.1 Executive Summary

1.1.1 Objectives

Carbon based nanomaterials (carbon nanofibers or carbon nanotubes) are promising candidate materials for reinforcing metallic matrices such as Al, Cu or Ti. The attractive mechanical properties (high mechanical strength) combined with interesting thermophysical properties (low coefficient of thermal expansion and high thermal conductivity) are of interest for many applications. However, most of the time, the beneficial properties of these nanomaterials are only exploited to a few percent. To take advantage of the excellent thermal and mechanical properties of carbon nanofibers or carbon nanotubes several challenges have to be solved at the same time.

- First of all the structure, quality and purity of the nanofiller material plays an important role. Depending on the targeted properties in the composite material either Multi-walled, Single Walled Carbon Nanotubes or carbon nanofibers are used as reinforcement or filler material.
- The dispersion of the nanomaterial in the matrix is another critical parameter that must be addressed.
- The compatibility/reactivity with the matrix must be considered, which depends on the chosen matrix material. While in case of titanium, reactions with the filler material are expected, in the case of copper poor wetting and adhesion between matrix and filler is observed. To take advantage of the mechanical or thermal properties of the filler/reinforcing materials, therefore, it is necessary to design and tailor the interface to overcome these limitations.

The main goal of INTERFACE has been to address these critical areas and to work on possible solutions to overcome these limitations and fabricate a carbon nanofibre reinforced Cu composite combining high thermal conductivity (> 400 W/mK) and a CTE in the range of 8-12 ppm/K. Such a material, which can be easily machined, has been tested as a heat sink in laser bars, LEDs and high power modules.

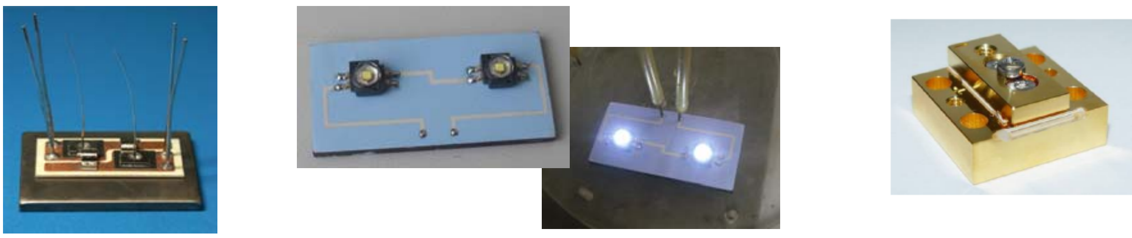


Figure 1: Overview of application tests on INTERFACE materials

To achieve this, the main objective of INTERFACE was divided into the following measurable sub-objectives:

- Fundamental study of the heat transfer at the copper/carbon nanofibre interface using flat models. Identification of possible interface modification strategies by introducing nanometre thick interlayers at the Cu-C interface, to optimise the mechanical and thermal interfacial properties. The aim is to reduce the thermal contact resistance of the interface below 10^{-8} m^2K/W (two orders of magnitude lower than the current state-of-the-art)
- To *functionalise* the surface of C nanofibres using the concepts developed in the flat model systems for which strategies to coat the nanofibres need to be developed.
- Understanding the role of interfaces in the thermo-mechanical properties of Cu MMCs reinforced with C nanofibers with the aid of computational simulations.
- Preparation of composite material with improved thermal properties: thermal conductivity >400 W/mK (two fold increase with respect to state of the art) at a reduced CTE of <10 ppm/K, incorporating the interfacial functionalisation achieved.
- First assessment of performance of the composite for different high thermally loaded applications

1.1.2 Proposed method

To achieve a two-fold increase with respect to the state of the art in the thermal conductivity of Cu composites reinforced with C nanofibres it is essential to reduce the thermal contact resistance at the interface by at least two orders of magnitude, by the incorporation of appropriate intermediates layer. In order to avoid a severe degradation of the used intermediate layers on the thermal conductivity of the copper matrix, the thickness of the intermediate layer must be kept as thin as possible (in the nanometer range). This demand in very thin layers requires suitable deposition techniques. Conventional electrochemical coating processes which have been used successfully for the coating of conventional carbon fibres will not be able to meet these requirements. The originality of the proposed work resides on using Physical Vapor Deposition (PVD), Chemical Vapor Deposition (CVD) and Colloidal Microwave Processing (CMP) techniques to functionalize the interface. Of course these methods must be adapted in order to provide uniform coatings of a well defined coating thickness. In several works the potential of these methods for coating of short fibres, particles or powders even of micron or submicron size has been shown either by PVD, CVD or CMP.

In order to identify suitable intermediate layers in combination with appropriate processing conditions it was necessary to employ methods which allow the structural study and the determination of the interfacial properties in a direct way, rather than from indirect methods as it is usually achieved. Therefore the project consisted was divided into two phases:

- In the first phase a fundamental study of the thermal contact resistance, mechanical strength and wetting behaviour of the Cu/C interface was conducted. The originality of the proposed work consisted on using flat model systems, made of thin layers of Cu and C, where the interface was directly studied, including the thermal transport. A comprehensive study of the influence of various pre-treatment/coatings of intermediate layers on the interface properties, the wetting behaviour and the conditions to avoid degradation of the constituents, was made using the flat model systems. The goal was to reduce the thermal contact resistance of the interface by two orders of magnitude with respect to the state of the art.

- The second phase conducted the knowledge transfer to a real composite material which was tested under application conditions. Three applications from various fields were identified:
  - Heat Sink Material for optoelectronics (LEDs)
  - Heat Sink Materials for Power Electronics (e.g. IGBTs)
  - Heat Sink and Electrical Contact Material for high-power diode lasers (HPDLs)

To achieve these goals, INTERFACE was divided into seven workpackages:

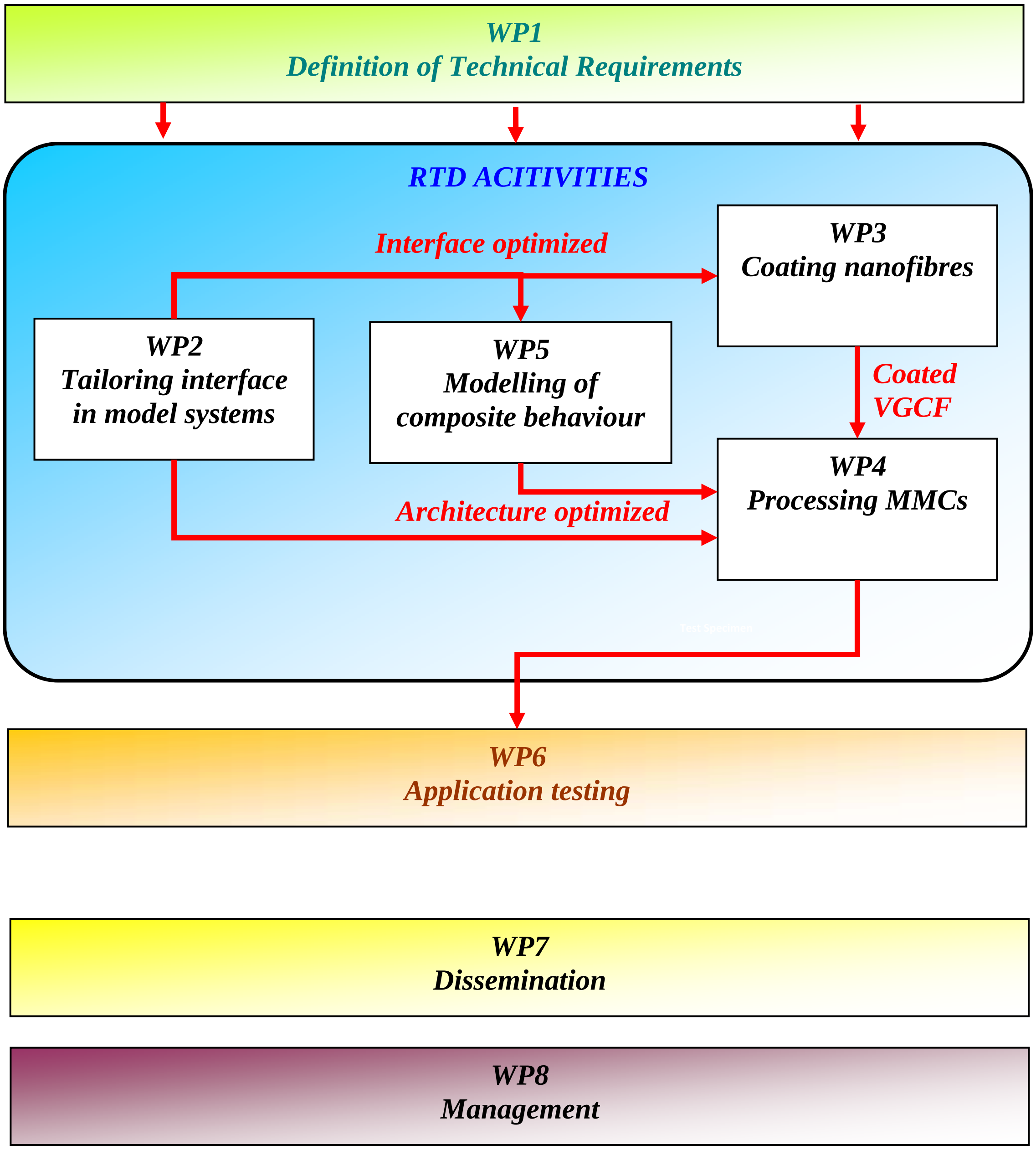


Figure 2: Overview of INTERFACE workplan

WP1: Definition of technical requirements
The objective of this workpackage was the specification of the end-users regarding material properties, size and shape. This definition includes the technical and commercial requirements for heat sink materials used for the selected applications.

WP2: Tailoring interface in flat model systems
The aim of this WP was to establish the interfacial systems that would enable the required thermal and mechanical behaviour in the Cu/C nanofibre MMC. To do this, Cu/interlayer/carbon 2-D test samples were fabricated and tested and analysed with respect to adhesion, thermal interface resistance as well as diffusion or dewetting during a heat treatment.

WP3: Functionalisation of nanofibres
The objective of this WP was the synthesis and coating / modification of carbon nanofibers in order to supply the fibres for the preparation of composite materials with tailored interfaces.
For functionalization, modification and coating, respectively, of the nanofibres, reliable mechanism for handling and dispersion of the particles have be identified.

WP4: MMC Processing and Characterisation
The objective of this WP was the preparation of composite materials from pre-coated VGCFs by using solid and liquid state processing as well as the characterisation of materials with respect to thermal, electrical, mechanical and microstructural properties.

WP5: Interfacial models for MMCs
The objective of this workpackage was to model the overall composite thermal and mechanical behaviour, in particular the composite architecture and the reinforcement volume fraction, in order to achieve the highest thermal conductivity, to reduce excessive residual stresses and to tailor the CTE of the material. In order to do that, modelling of the interfaces in terms of load and heat transfer has been a critical issue, as well as using authentic data and process influence supplied by workpackages WP2 and WP4.

WP6: Application Testing
The objective was to manufacture first tests parts with the defined geometries from WP 1 and to make first application tests for the different applications. For this, LEDs, laser diodes and high power devices were mounted on the fabricated Cu carbon nanofiber composites and tested according to standard tests (thermal cycling, different power etc.)

WP7: Dissemination
The goal was to ensure the widest possible dissemination of the project achievements and activities and the exploitation of the expected results.

- The basic activities of publication of journal papers and trade articles where appropriate.
- Implementing and maintaining the plan for using and disseminating knowledge
- Setting up a dedicated webpage, with internal and external faces, for the promotion of research results, information of the interested general public as well as communication among the members of the Consortium
- Filing of patents in case new material compositions, processes or new products with high potential are developed

The following table gives an overview on the project partners involved in the INTERFACE project including their nationality and short names.

http://www.ceit.es/interface

Partic N.	Participant name	Short name	Country
1	*Centro de Estudios e Investigaciones Técnicas de Gipuzkoa*	CEIT	Spain
2	*AIT – Austrian Institute of Technology*	AIT	Austria
3	*Institut fuer Niedertemperatur-Plasmaphysik e.V. Greifswald*	INP	Germany
5	*Ruhr-University Bochum*	UBO	Germany
6	*The Chancellor, Masters and Schollars of the University of Cambridge*	UCAM	United Kingdom
7	*Linkopings Universitet*	LIU	Sweden
8	*Centre Riserche Fiat Societá Consortile per Azione*	CRF	Italy
9	*Future Carbon GmbH*	FC	Germany
10	*IMA Engineering Services Limited*	IMA	Malta
11	*Jenoptik Laserdiode GmbH*	JOLD	Germany
12	*Fundación IMDEA Materiales*	IMDEA	Spain

1.1.3 Main achievements

The main achievements of the project are summarised below with respect to the state-of-the-art and the industrial objectives of the end-users:

- **Engineering the Cu-C interface towards higher thermal conductivity and better adhesion**

LIU fabricated different Cu/interlayer/carbon 2-D test samples using PVD (Physical Vapor Depositon) (Figure 3 and Figure 4), with the objective of carrying out fundamental studies of thermal conductivity and mechanical strength of the engineered interfaces. In particular, Cr and Ti interlayers with and without plasma treatments were tested, leading to the following results:.

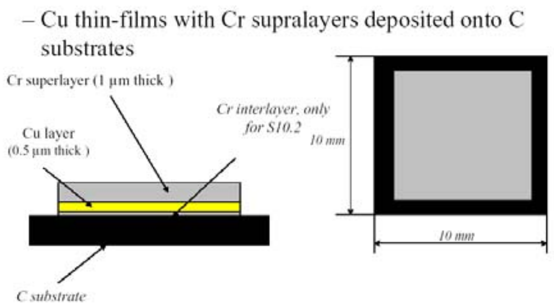


Figure 3: Overview on sample composition/size

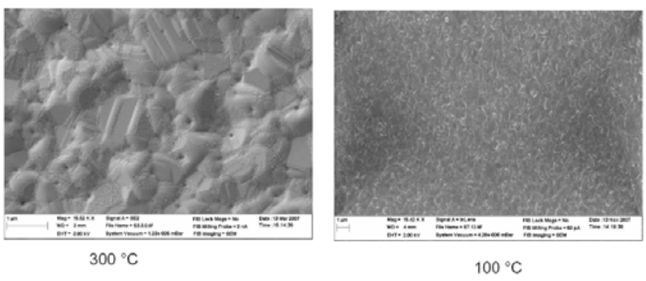


Figure 4: Copper coatings (PVD) deposited at different substrate temperatures

- Together with partners CEIT and AIT, the mechanical adhesion strength of the interfaces was tested by pull-off measurements, cross sectional nanoindentation and supralayer indentation tests. Of all three, the last method was found to be better suited to test this type of samples and to obtain information on the adhesion strength of the coating on the substrate. Here the indentation induced delamination extent was used to derive the adhesion strength, supported by FE modelling (Figure 5). Using this method, it was concluded that a 2.5 nm thick Ti interlayer can induce a 3-fold increase in adhesion strength, while a 10 nm thick interlayer resulted on the impossibility of inducing delamination of the Cu film. Similar results were found for Cr interlayers.

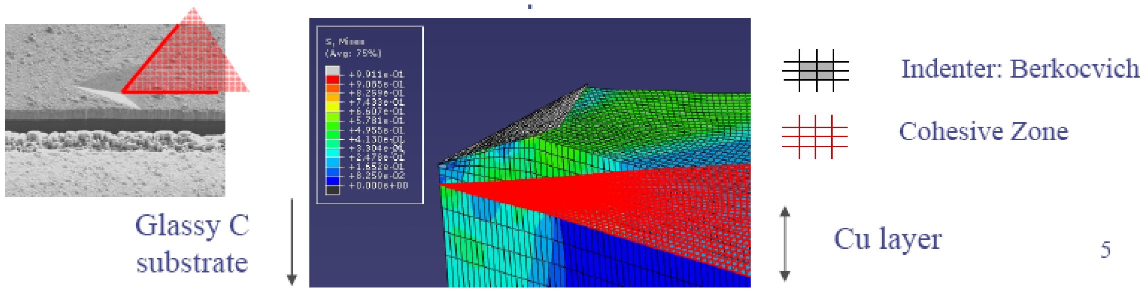


Figure 5. Modelling top indentation tests.

- As an important outcome of the project, UBO, in collaboration with the University of Reims, has developed novel metrology techniques to assess the interfacial contact resistance in flat systems, using various photothermal methods. The task has resulted specially challenging for the glassy carbon used as substrate material in the flat model systems, but investigations carried out in parallel on diamond substrates have shown that the high sensitivity of the methodology. By investigating different metal-substrate systems with different measurement devices available at Bochum (from 10 Hz to 10kHz) and Reims (100 to 100kHz), supported by phase/amplitude simulations for Cu films on glassy carbon substrates and diamond substrates, it was possible to establish the measurement sensitivity of the thermal contact resistance (TCR). For the glassy carbon substrate, TCRs below 10^{-7} m^2W/K could not be measured, while for diamond substrates, the technique can measured TCRs as low as 10^{-8} m^2W/K could be found. Figure 6 shows the phase shift for different frequencies.

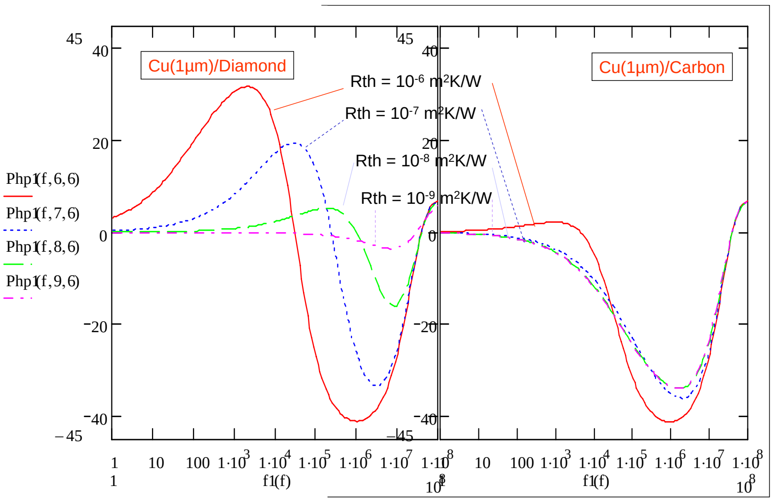


Figure 6. Phase shift for 1 µm Cu coating on diamond and glassy carbon substrates as a function of the modulation frequency

- In addition, thermal stability studies show that the intermediate layers prevent dewetting of the Cu layers , further showing the benefit produced.

- Therefore, the main outcome of the fundamental studies, is that the intermediate layers (Ti, Cr) can induce an **improvement** of at least two orders of magnitude in the **thermal interface conductance** (reaching the limit of the metrology used in carbon glassy substrates, 10^7 W/m^{2}K) and a **three-fold increase in the adhesion strength**.

- **Functionalisation of carbon nanofibres**

Partner FC produced different types of VGCFs for the project: 3 types of carbon nanotubes and fibres were used as basic material for the project as well as 2- and 3-dimensional structures for later-on infiltration processes. The synthesis of VGCFs was carried out to support all partners with needed amounts of materials. Also 2- and 3-dimensional structures of VGCFs were developed and brought into the project as well as coated grades (coated by CMP process, a microwave based coating process) and dispersions as shown in Figure 7. Using this, the following functionalisations were achieved:

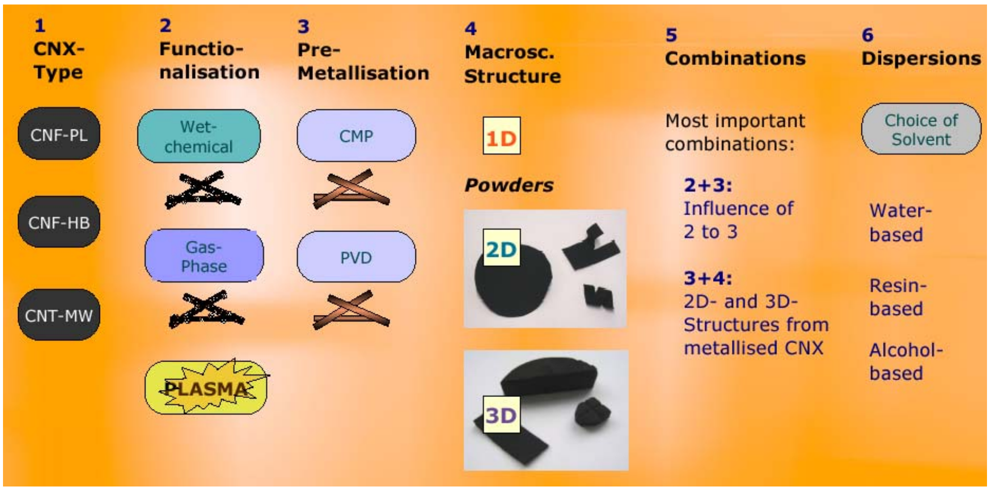


Figure 7: Overview on different carbon nanomaterials and structures which are developed by FC

- In order to transfer the knowledge developed in the flat model systems to the carbon nanofibres, partner INP built up a PVD coating device for the coating of carbon nanofibers with different elements (Cr, Ti). The process is based on the physical vapour deposition method (see schematic overview in Figure 8 and experimental set-up in Figure 9). The Ti coating leads to spontaneous self-combustion in contact with air and therefore, all efforts were conducted to use Cr interlayers, that showed clear benefits in the flat model systems. The Cr deposition process was optimized to reduce the amount of reaction products (carbides, oxides). The optimised process was used to produce coated CNFs in sufficient amount to fabricate prototypes of composite materials to be tested in the final applications. In parallel, FC developed a colloidal microwave process to metallise nanofibres with different elements.

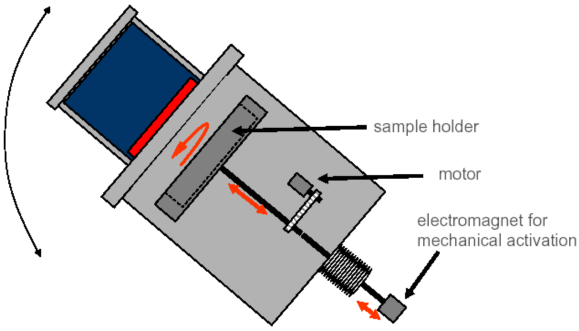


Figure 8: Geometric arrangement of magnetron sputter device

Figure 9: Experimental set-up for coating of CNFs

- **Fabrication of CNF reinforced Cu composites**

Two different techniques were followed for MMC processing by AIT, LIU, IMA and CEIT: solid state and liquid phase processing. For solid state processing, three different routes have been investigated:

- Mixing of Cu powders+alloying elements with carbon nanofibers
- Mixing PVD coated CNFs with copper powder
- Using electrochemically Cu coated CNFs combined with admixing of Ti or Cr.

In the case of the liquid phase processing route, it was found that wetting of the carbon nanomaterials with the copper alloy is possible, e.g. by the addition of Cr. One of the main problems arising from the addition of third elements is the reaction with the CNFs. Even after a further reduction of the contact time during the infiltration, it was not possible (only some tens up to some hundreds of µm could be infiltrated) to achieve an infiltrated bulk material for further measurements and thermal characterisation. Figure 10 shows the cross section, with a severe reaction between the Cr addition and the CNF. Therefore, efforts were directed to solid state processing techniques.

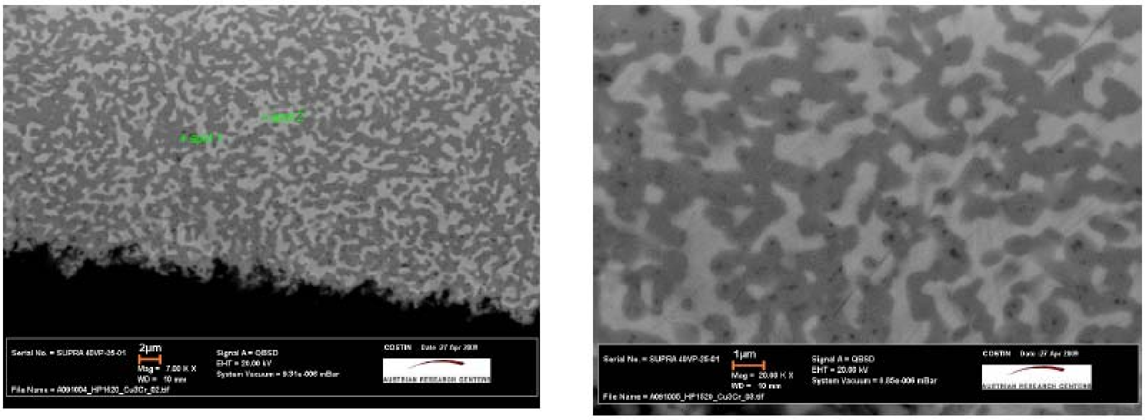


Figure 10: Microstructure/Cross section after infiltration experiment.

The main achievements of the processing studies can be summarised as follows:

- It was found that a high temperature pretreatment of the FC CNFs at 2750ºC improve the final thermal diffusivity of the final composites.
- Processing routes based on admixing Cu powders with CNFs produce unsatisfactory dispersion of the CNFs on the final composites.
- The electrochemical Cu coating method resulted in a very good process to obtain composites with a good dispersion of the carbon fibers.
- It was found that the Cr PVD pre-coating of the CNF showed a better coat ability compared to the naked fibers. Even a rather low amount of coating (thickness of some nanometer) has a positive effect on the electroless coating process (see Figure 11 and Figure 12).

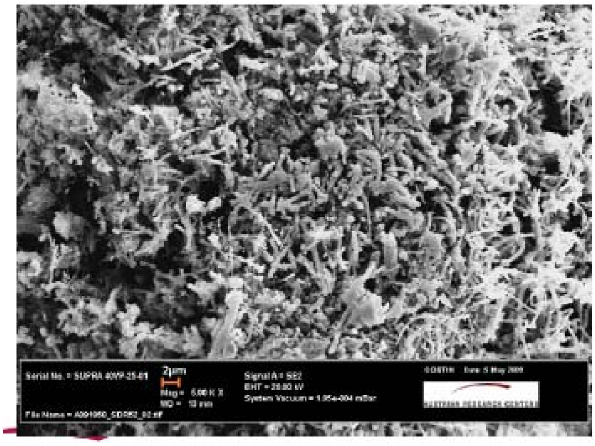


Figure 11: Electroless Cu plating of nanofibres.

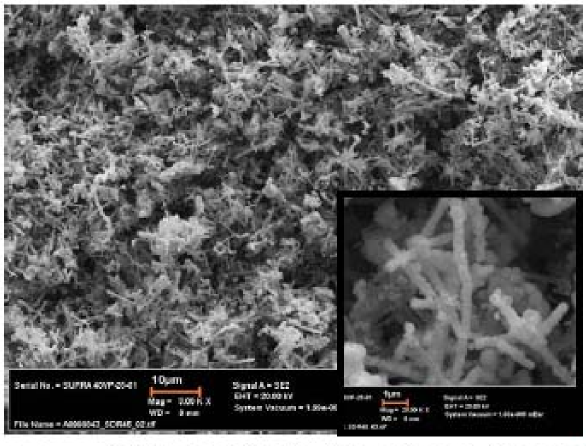
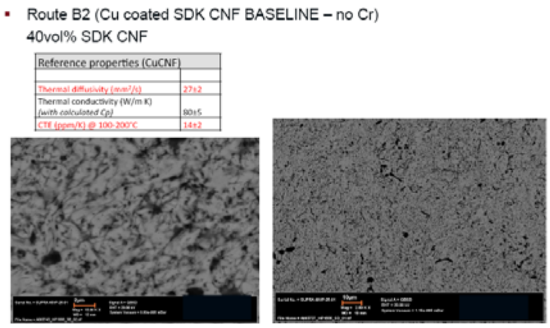


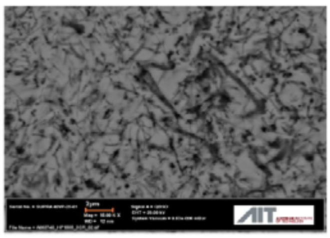
Figure 12: Electroless Cu plating of nanofibres with a PVD Cr coating.

- For the preparation of bulk materials, the coated powders were consolidated using hot-press. All the materials were characterised with respect to their thermal properties. An intensive microstructural analysis was carried out to verify the homogenous dispersion of the CNFs (Figure 13 and Figure 14).



Reference properties (CuCNF)	
Thermal diffusivity (mm²/s)	27±2
Thermal conductivity (W/m K) *(with calculated Cp)*	80±5
CTE (ppm/K) @ 100-200°C	14±2

Figure 13: Overview on thermal properties an microstructure of composites made by Route B2 (SDK fibers coated by electroless deposition with copper)



Properties (CuCNF8Cr) 0.9v%Cr	
(CNF Cu coating by IMA)	
Density SDK (g/cm³)	2
Density composite (g/cm³)	6.6±0.1
Thermal diffusivity (mm²/s)	43±5
Specific heat capacity (J/kg K) *calculated by rule of mixture*	433,2
Thermal conductivity (W/m K)	122±16
CTE (ppm/K) @ 100-200°C	14±2

Properties (CuCNF11Cr) 1.33v%Cr	
Density composite (g/cm³)	7.1±0.2
Thermal diffusivity (mm²/s)	35±4
Specific heat capacity (J/kg K) *calculated by rule of mixture*	432,8
Thermal conductivity (W/m K)	108±15
CTE (ppm/K) @ 100-200°C	14±2

Figure 14: Overview on thermal properties an microstructure of composites made by Route D1 (Cr. PVD coated SDK fibers followed by electroless Cu deposition)

- A two-fold increase in thermal conductivity has been achieved for the PVD Cr coated CNFs compared to the baseline process – both are characterized by a good dispersion of the CNFs – but still the thermal conductivities and CTEs are below the expectations and the results from simulations.

- Motivated by the relatively low thermal conductivities obtained with respect to expectations, a thourought investigation was carried out by UCAM. Generally, in the literature, the view is that this is caused by the poor interfacial thermal conductance. While this effect is no doubt important, it was observed that the VGCFs lose their graphitic structure during processing and that this is the predominant reason for the fall in thermal conductivity to that of copper containing an equivalent volume fraction of pores. Extensive transmission electron microscopy has been carried out to characterise this behaviour. A technique based on Raman spectroscopy has also been developed to quantify this effect. This allows a study of many of the important details much more rapidly and this is being used elsewhere in the programme. It is found that similar behaviour occurs in K1100 8µm carbon fibres. However being larger the amorphous region does not penetrate throughout the fibre. Although some amorphization occurs on heating, the primary effect appears to be associated with the wetting aids that are added to improve the interface conductance diffusing into the nanofibre.

- UBO/Reims carried out some additional work on the measurement of the specific heat on composite materials as well as to determine the anisotropy of material properties as a consequence of the CNF alignment during the processing. An anisotropy factor of around 100 was measured, with a x-y thermal conductivity of more than pure copper. The main drawback in this case is that probably the x-y thermal conductivity required by the enduser can be met but on the expense of the thermal conductivity in z-direction which would be below 10 W/mK. Such material performance could be of relevance in other applications where such anisotropy is of advantage.

- **Modelling the thermal conductivity of CNF reinforced Cu composites**

CEIT, IMDEA and UCAM have developed several models to understand the effect of CNF distribution, CNF properties and interfacial properties on the final properties of the composite materials.

- CEIT and IMDEA have developed tools to simulate the thermomechanical behaviour of short fibre reinforced composites by the Finite Element Method (FEM). A Unit Cell Approach has been followed, by which the composite is simulated as a periodic repetition of a Representative Volume Element (RVE), containing a small number of nanofibres, as shown in Figure 16.

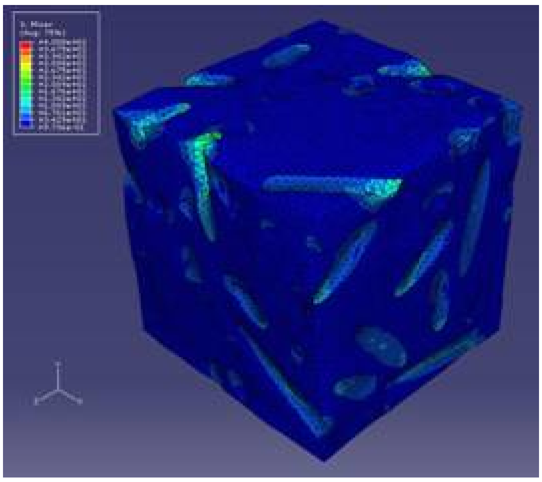


Figure 15. 3D randomly oriented fibres (30%) embedded in the matrix subjected to a temperature increase.

- A good agreement of the FE simulations with analytical models developed by UCAM has been obtained. The established numerical model allow a variety of parameter variations such as architecture (2D/3D random), aspect ratio, fiber arrangement, including of imperfect interface (partial debonding etc), as shown in Figure 16.

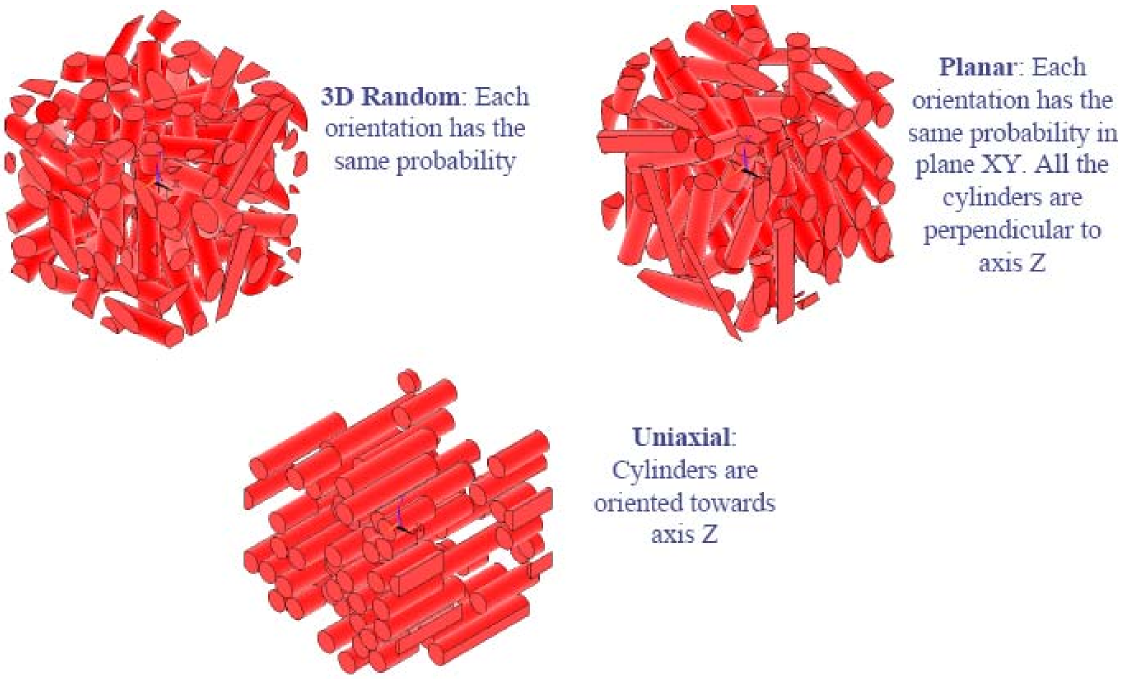


Figure 16: Different unit cells designed for numerical analysis

- In the following figures the results of the achievable thermal properties for each of the CNF arrangements are shown.

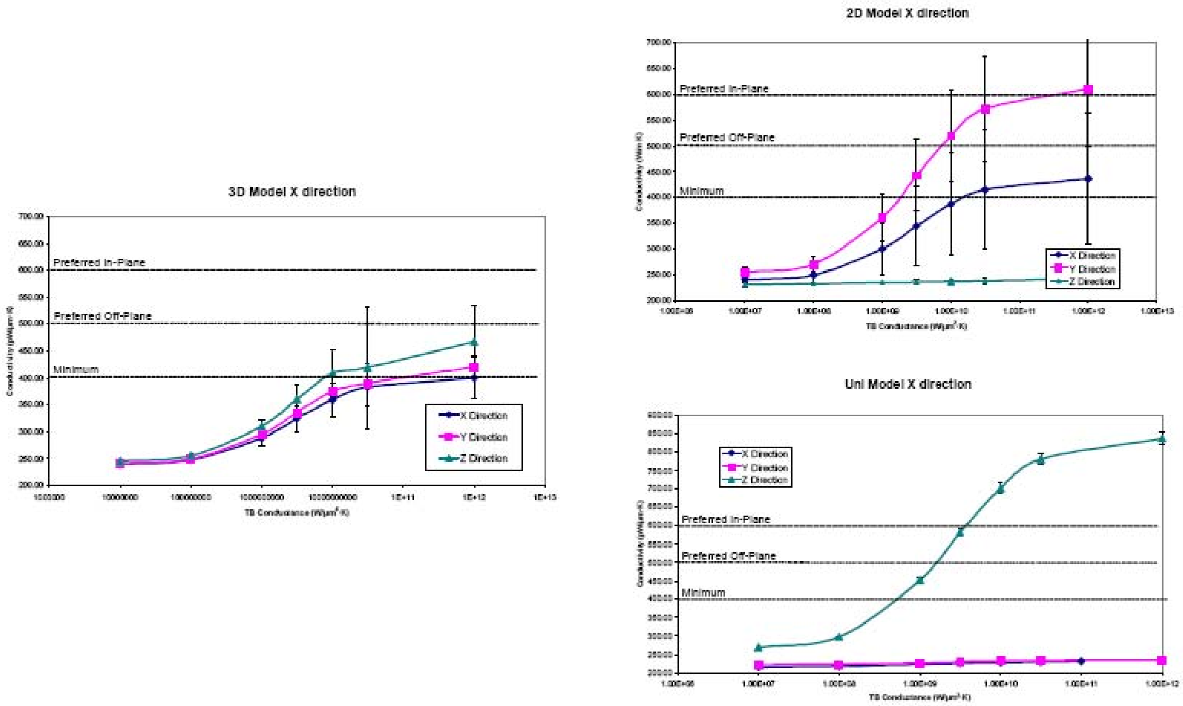


Figure 17: Thermal properties for different CNF alignment as a function of the Thermal Boundary Conductance

- Assuming a 3D random arrangement and in order to obtain a thermal conductivity of >400 W/mK, a thermal boundary conductance (inverse of the TCR) of 10^{10} W/m^{2}K would be required. Due to the anisotropy of the CNFs, with a very low thermal conductivity in the transverse direction, a preferential alignment of the CNFs induces a large anisotropy of the composite material. Taking into account that the experimental thermal boundary conductances measured were of the order of 10^7 - 10^8 W/m^{2}K, the expected thermal conductivities would be around 250-300 W/mK, lower than initial expectations. This is even pronounced if a planar preferential orientation is assumed, in concordance with the experimental observations, in which case a especially poor thermal conductivity in z-direction is expected. The modeling results are in agreement with the experimental observations and underline some of the factors that contribute to the thermal conductivities obtained in the final composite, lower than expectations, even for large improvements of the thermal contact resistance.

- **Application testing**

The composites have been tested against three different applications by the end-users:
- Heat Sink Material for optoelectronics (LEDs)
- Heat Sink Materials for Power Electronics (e.g. IGBTs)
- Heat Sink and Electrical Contact Material for high-power diode lasers (HPDLs)

By using the most favourable compositions various prototype materials were prepared to test the material in the three different applications. Examples of the prototypes are shown in Figure 18.

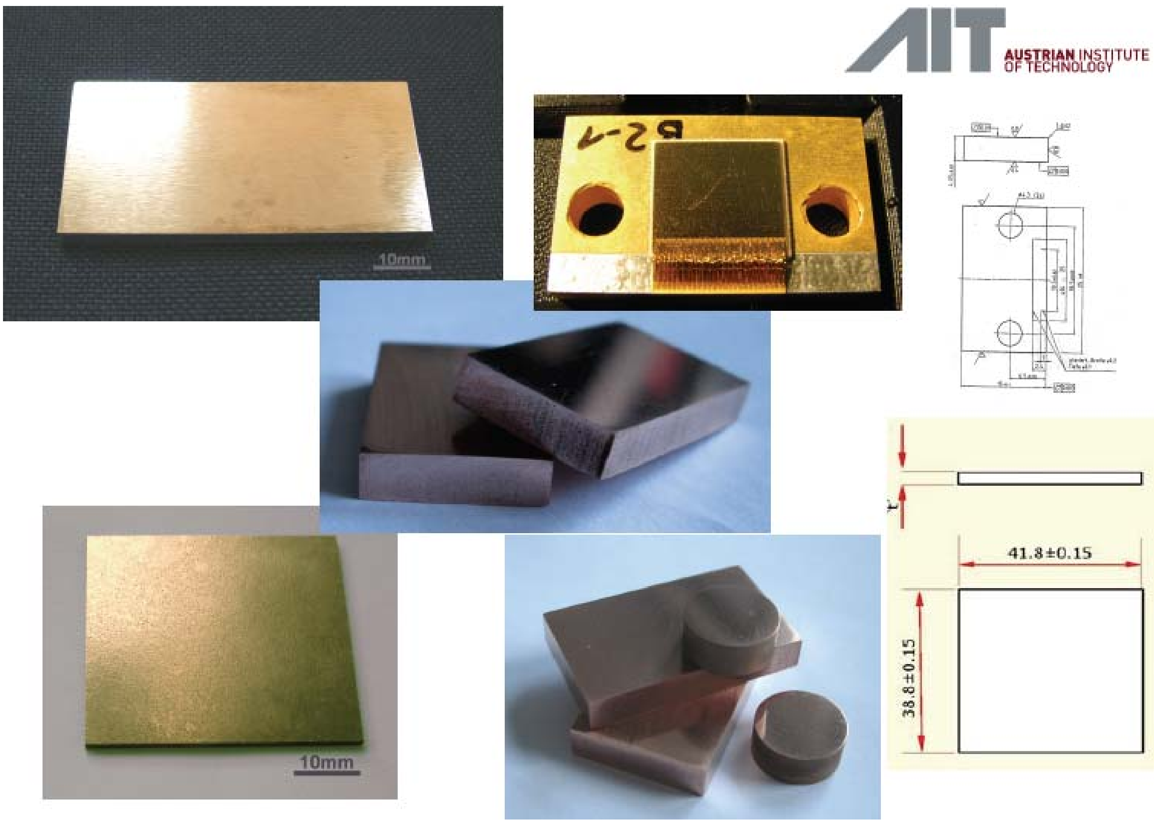


Figure 18: Prototypes manufactured according to the specification of the 3 endusers.

The main conclusions of the application testing can be summarized as follows:

- **LED APPLICATION:** The standard fiber board works up to 3 W. With two materials provided by the INTERFACE project an improved performance compared to the standard Al solution was achieved. The LEDs could be operated up to almost 6 W.

- **LASER DIODE APPLICATION**: The requirements for this application, very demanding, cannot be achieved, although some benefits related to assembly of the heat sink were identified, as summarised below:
  - High thermal conductivity (higher than that of pure copper) could not be achieved
  - Coefficient of thermal expansion close to the used heat source material was not obtained.
  - High electrical conductivity to enable high electrical current conduction was achieved
  - Good bulk and surface machinability
  - Possibility of coating by galvanic/ electroless plating and PVD/ CVD
  - Possibility of direct soldering

- **POWER SWITCHING APPLICATION**: The conclusions of the tests for this application are:
  - Results indicate that the composites with PVD Cr interface show the best performance among the INTERFACE materials. Here a significant improvement of the designed interface (Cr coating) compared to the uncoated CNF is visible, but all tested MMC samples gave a higher thermal resistance (Rth - steady state) to that of copper.
  - Higher Rth than expected might be due to the depression observed in various base plates.
  - The number of samples for testing was too small to draw an accurate conclusion at this stage.

2 Dissemination and use

2.1 Scientific Publications

The following is a list of the INTERFACE technical papers published:

- E. Neubauer, M.Kitzmantel , C. Eisenmenger-Sittner, I. Smid, P. Angerer, *Copper based composites reinforced with carbon nanofibres,* Proceeding of the 2007 International Conference on Powder Metallurgy & Particulate Materials, POWDERMET 2007, May 13-16, Denver, Colorado
- E. Neubauer et al., Pulvertechnologische MMCs, ARC Workshop on “Nanomodified Materials”, 5-6. September 2007, Vienna Austria
- R. Nagel, et. al., *Copper based composites reinforced with carbon nanofibers,* CERN Workshop on “Workshop on Materials for Collimators and Beam Absorbers , 3-5. September 2007, Geneva, CH
- Francisco Macedo, Filipe Vaz, Ana C. Fernandes, Luís Rebouta, Sandra Carvalho, Klaus H. Junge, Bruno K. Bein**,** *Thermal Characterization of Hard Decorative Thin Films,* Plasma Processes and Polymers 4, S1 (2007), S190-S194
- Francisco Macedo, Attila Gören, Ana C. Fernandes, Filipe Vaz, Juergen Gibkes, Klaus H. Junge, Jean L. Nzodoum-Fotsing, Bruno K. Bein, *Potential of Modulated IR Radiometry for the On-Line Control of Coatings*, Plasma Processes and Polymers 4, S1 (2007), S857-S864

- M. Kitzmantel, C. Eisenmenger-Sittner, D. Cunningham: "*Nanofeatured Carbon-Copper Metal Matrix Composites*"; Poster: 2008 Junior Science Conference, Wien; 16.11.2008 - 18.11.2008; in: „Proceedings of the Junior Science Conference 2008“, H. Kaiser (Hrg.), R. Kirner (Hrg.), (2008), S. 179-180
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2.2 WEB news

The **INTERFACE Project website** can be found at http://www.ceit.es/interface

2.3 Major planned events

- **The INTERFACE consortium members** organised a special symposium on C-Cu composites and other materials for heat sink applications (B17 - Metal Matrix Composites Reinforced with Nano-sized Reinforcements) at the major international conference E**UROMAT** in Glasgow in 2009 (Euromat 2009). The event was a success, with over 19 papers presented orally and 20 poster presentations. The major results presented in the symposium will be published in a special issue of Composite Science and Technology, the journal ranked 1st in the area of Composite Materials, which is in preparation.

2.4 Patents filed

- Patent application DE 10 2007 051 570.9 “Verfahren zur Herstellung eines Verbundwerkstoffs, sowie Verbundwerkstoff,Verbunwerkstoffkörper und Aschlussvorrichtung” has been put forward by JOLD, ARC and FC