The International Energy Agency
Implementing Agreement on Advance Materials for Transportation Applications

Annual Report to IEA Secretariat

For Calendar Year 2010

AMT Executive Committee

Jan 15, 2011

Summary

Management and organization

In the last two years, AMT Executive Committee (Exco) has systematically implemented the new operating plan put in place in 2009. Australia became an official AMT member on May 6, 2010. The Shanghai Institute of Ceramics also became a member of AMT as the second Contracting Party from China in addition to Lanzhou Institute of Chemical Physics. VTT of Finland, Technion University of Israel, and the Metals Research Institute in Shenyang, China are at various stages of applying as contracting parties to join AMT.

With the addition of new members, AMT's technical activities are moving forward rapidly. The new Annex VIII on thermoelectric materials has conducted two international round robin studies since its inception a year ago. Results on measurement of thermal conductivity and Seebeck coefficients (an indication of thermal electric power efficiency) showed large variations in the measurements, hampering the development of new thermoelectric materials. This has attracted much international attention. Annex VII is sponsoring a round robin study on nanomaterials, a new class of low cost high performance materials that is increasingly important in light-weighting and friction reduction. The Exco meeting was held in Shanghai, China for the first time in June, 2010, hosted by the Shanghai Institute of Ceramics. We look forward to welcome more participants in the region.

Technical activities

Significant progress was made during 2010 by the five annexes. These are detailed in the attached annex reports. Two new annexes were activated this year and they are making significant progress in moving forward. Annex VII on the development of measurement techniques for nanomaterials has completed test method development and is ready to launch the round study. Annex VIII on thermoelectric materials has completed two preliminary round robin studies on thermoelectric materials in the first year of its operation.

The transportation technologies are undergoing rapid evolution as a result of the world wide consolidation in the automobile industry during the last two years. New technologies on electric cars, plug-in hybrids, and fuel cell propulsion are being introduced, at the same time, the new CAFÉ standard require reaching 35 mpg by 2020 is prompting rapid development in fuel economy improvements. Materials play a pivotal role in reaching the new CAFÉ standards. AMT activities focus on light-weighting and friction reduction through materials are central to the next generation fuel efficient transportation technologies.

INTERNATIONAL ENERGY AGENCY
CO-OPERATIVE PROGRAMME ON ADVANCED MATERIALS FOR TRANSPORTATION APPLICATIONS (IEA-AMT)

Annex IV Integrated surface technology for friction reduction in engines

Covering the period: Jan 1, 2010 to Dec. 31, 2010

1. **Title of Annex:** Integrated surface technology for friction reduction in engines

2. **Annex Participants**:

China:	Dr. Junyan Zhang, Lanzhou Institute of Chemical physics
USA:	Dr. Stephen Hsu, George Washington University
Australia:	Dr. Gwidon Stachowiak, University of Western Australia

3. **Annex Objectives**

The objectives are: to develop friction reduction surface technology by task-sharing of duties; share information; develop test methods; develop surface characterization methods for textured surfaces. The integration of surface technology include the following shared tasks: texture design (US), protective thin films (diamond like coating, DLC) (China), and surface description techniques (Australia). When the need arises, round robin studies may be conducted to share information and develop precision statement on test methods for international standards.

4. **Technical Progress and accomplishments for the reporting period**:

This annex is a task-shared activity with the scope of work as follows:

Subtask 1: Technical Information Exchange (ongoing and at every meeting)
Subtask 2: Characterization/description of integrated surface technology
Subtask 3: Performance evaluation of integrated surface technology

Planned milestones:

2010 Assessment of the effectiveness of conventional 2-d roughness parameters such as Ra, Rq, Rp, Rv, Rt in describing textured surfaces; Characterization of friction films and bonded chemical films on textured surfaces; Assessment of various DLC films for their suitability in engine applications

2011 Development of 3-d descriptors of discrete textured surfaces; Assessment of various bench test procedures simulating engine friction; finalize the DLC film specifications and surface chemistry

2012 Conduct motored engine tests and/or engine tests and correlate the results with bench tests; Develop final design guidelines for integrated surface technology for engine applications

Major accomplishments in 2010:

Subtask 1: Information exchange (US, China, Australia)

Technical reports on the progress of the annex were presented during the two technical symposia held before the Exco meetings in Shanghai, China (June 2010) and Oak Ridge National Lab, US (Nov. 2010). Results were shared by visits (US visited Lanzhou in September, 2010) and an annex meeting held in Perth, Australia in conjunction with the AsiaTrib in December 2010.

Subtask 2: Characterization of textured surface (Australia)

Surface characterization methods currently in use were developed for 1D surface profiles, i.e. they work well with isotropic surfaces exhibiting the same features in all directions. However, problems arise with the anisotropic surfaces which exhibit different features in different directions, i.e. they cannot be effectively characterized. The typical examples are the surfaces with directionally aligned grooves or discrete geometric dimples, e.g. In addition the surface topographies are multi-scale objects, i.e. exhibiting different length scales of surface features. Therefore any new surface characterization methods developed must allow for this multiscale effect.

A concept of a self-transformability, meaning that one part of the surface image can be transformed into another part of the image reproducing itself almost exactly, was used to encapsulate entire surface topography information into the set of mathematical transformations, i.e. in the fractal model. If, for example, such a model is constructed for a textured surface it will contain the detailed information about, the dimple shape, dimple spacing, size, orientation, etc. [1-4]. The fractal model constructed called a Partition Iterated Function System (PIFS), using set of N contractive affine transformations, i.e. $\mathrm{PIFS} = \bigcup_{j=1}^{N} f_j\left(DOM_j\right)$ [1-3]. Each transformation f_j converts a larger part of the surface (called domain) DOM_j into a smaller part (called range) RAN_j located elsewhere on the same surface, i.e. $f_j\ (DOM_j)=RAN_j,\ j=1,2,3,\ldots,N$. When an arbitrary image is applied iteratively to the PIFS, a sequence of decoded images (called intermediate images or transition frames) converging to attractor is obtained. Details are described in [1-5]. A combination of fractals and wavelets was used to account for the effect of multiscale nature of surface topographies, i.e. wavelets are used to decompose the surface image containing 3-D surface topography information into different scale components, e.g. roughness, waviness, form, etc., while fractals are used to characterize the surface topography over the achievable range of scales. [1-5]. Recent work on the multi-scale characterization of surface roughness and anisotropy and application of PIFS to surface description will be published in [8,9].

Current approach to the optimization of surface texture shapes in lubricated contacts is, in most cases, by ‘trial and error’, i.e. changes are introduced and their effects are studied. We attempt to develop a systematic computational approach for the texture shape optimization aiming at reducing friction and increasing load capacity in hydrodynamic contacts. This new approach to be developed would be based on control parametization and parameter selection originally developed for control systems governed by ordinary differential equations (ODEs) so that it can be used for non-linear partial differential equations (i.e. the governing

Reynolds or Navier Stokes equations that describe the lubricant's flow and pressure in hydrodynamic contacts.

Our initial study showed that this approach works for 1D cases for which the Reynolds equation can be transformed into a set of ordinary differential equations (ODEs). In this study, stepped slider and journal bearings, a tilted pad bearing and a partially textured infinitely long parallel bearing were optimized for the maximum load capacity. Geometry of the partially textured parallel bearing was optimized for the maximum load carrying capacity. The optimal ratios of $\varepsilon_{opt}=1.52$ and $\xi_{opt}=0.15$ were calculated for the bearing textured with two dimples $m=2$ and the untextured portions of $\ell_0=0.5$ and $\ell_1=0.125$ [6]. The results obtained agree with data published in [7].

Subtask 3: Performance evaluation of integrated surface technology (US, China)

To prolong the life of textures in engines, wear protection has be enhanced. Last year, we evaluated several commercial diamond-like carbon thin films that increased the life of textures under a durability test protocol of cyclic sliding under various loads and speeds. This year, China contributed their newly developed hard and tough DLC thin films. The life of the textures was enhanced further.

Since DLC films do not react with conventional lubricant antiwear additives and their active sites are far less than those of metals, we began to search for novel chemistry that will react with DLC films and form sufficient chemical film density to protect the DLC and at the same time, impart further friction reduction properties.

DLC coated samples were used to test various chemistries. A pin-on-disk step loading test procedure was used to evaluate the chemistry. The load was varied from 1 N to 25 N, and the speed was varied from 0.015 m/s to 0.20 m/s to cover the hydrodynamic and boundary lubrication regimes. To extend the load range of the test, two diameters of the pin (balls) were used (1.5mm and 6.125mm). At each sliding speed, 8 different loads were applied and the steady state friction for that film was recorded.

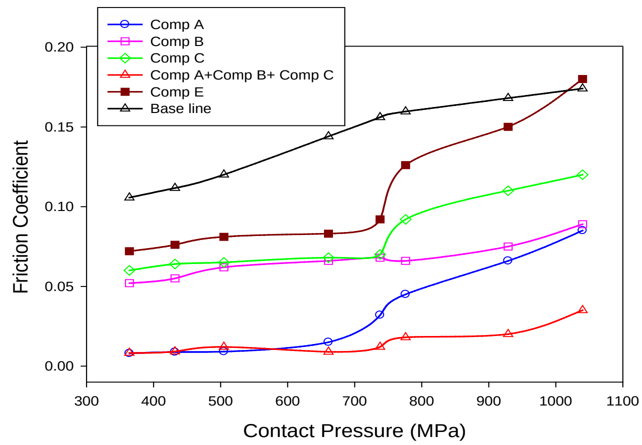


Fig. 1. Frictional characteristics of chemical films as a function of contact pressures

As can be seen in Fig. 1, friction drops from boundary lubrication conditions (0.08-0.15) to 0.03, typical of hydrodynamic lubrication regime. Mixed compounds appear to be able to control the friction over the speed and load range much better than a single compound. These chemistries were then combined with textures, DLC films, and tested in the durability test sequence. The life of the system was extended ten times.

5. Exchange of Information during the reporting period:

See answer under subtask 1 above.

6. Technical Objectives: Is the project meeting the technical objectives as outlined in the Annex plan?

Yes, the project is meeting the objectives as outlined in the Annex plan (updated in the Operating Plan)

7. Changes to Annex scope or objective:

The annex scope and objective have been updated in the current Operating Plan. With the addition of Australia, the Annex has been organized into task shared cooperation in which each country takes a lead over a major technical area and sharing results.

8. Publications:

1 P. Podsiadlo and G.W. Stachowiak, Scale-invariant analysis of wear particle morphology - a preliminary study, Tribology International, 33 (2000) 289-295.

2 P. Podsiadlo and G.W. Stachowiak, Scale-invariant analysis of wear particle morphology. I: Theoretical background, computer implementation and technique testing, Wear, 242 (2000) 160-179.

3 G.W. Stachowiak and P. Podsiadlo, Surface characterization of wear particles, Wear, 225-229 (1999) 1171–1185.
4 P. Podsiadlo and G.W. Stachowiak, Scale-invariant analysis of wear particle surface morphology. II: Fractal dimension, Wear 242, (2000) 180-188.
5 G.W. Stachowiak and P. Podsiadlo, 3-D characterization, optimization and classification of textured surfaces, Tribology Letters, 32 (2008) 13-21.
6 A. Guzek, P. Podsiadlo and G.W. Stachowiak, A Unified Computational Approach to the Optimization of Surface Textures: One Dimensional Hydrodynamic Bearings, Tribology on Line, Vol. 5, No 3, 2010, pp. 150-160.
7 R. Rahmani, A. Shirvani, H. Shirvani, Optimization of partially textured parallel thrust bearings with square-shaped micro-dimples, Tribology Transactions, 50 (2007) 401-406.
8 M. Wolski, P. Podsiadlo, and G.W. Stachowiak, Applications of the variance orientation transform method to the multi-scale characterization of surface roughness and anisotropy, Tribology International, accepted, 2010.
9 Wolski, P. Podsiadlo, and G.W. Stachowiak, Effects of information loss in texture details due to the PIFS encoding on load and friction in hydrodynamic bearings, submitted to Tribology International, 2010.

INTERNATIONAL ENERGY AGENCY
CO-OPERATIVE PROGRAMME ON ADVANCED MATERIALS FOR TRANSPORTATION APPLICATIONS (IEA-AMT)

Annex V: Advanced Corrosion Protection Technologies for Structural Magnesium Alloys Used in Transportation Industry

Covering the period: 1/1/2010 to: 12/31/2010

1. Title of Annex: Advanced Corrosion Protection Technologies For Structural Magnesium Alloys Used In Transportation Industry

2. Annex Participants:

Canada:	Wenyue Zheng, MTL, Canada (Lead investigator, Derushie, Roy, Bibby)
	Joey Kish, McMaster University, Canada
	J. Villafeurte, Julio, Centre-Line Ltd, Canada
	J. Jeckle, Meridian technologies Ltd, Canada
	G. Song, GM Research, Canada, USA
USA:	Tim Skszek, COSMA Engineering, USA ,
China:	B. Jiang, Xian Univ. of Tech, China
	En-hou Han, IMR-CAS, China
Germany:	W. Dietzel/C. Blawert, GKSS, Germany

3. Annex Objectives:

The objective of this Annex is to develop advanced coating technologies that can prevent stress corrosion cracking (SCC) and galvanic corrosion for magnesium alloys, and to establish the threshold loading (stressing) conditions for representative wrought structural products (AZ31, AM30, ZK alloy). The project aims to advance our understanding of the micro-mechanisms governing SCC of Mg alloys in typical automotive in-service environment, which is essential for successful technology development.

4. Technical Progress and accomplishments for the reporting period:

Two tasks were conducted in 2010:

Task II: Test specimen preparation and coating

Task III: Stress corrosion cracking testing.

Task II test specimen preparation and surface coating.

II-1: Anisotropy effect: Wrought products (AZ31, AM30) were machined into test specimens and the microstructure of AZ31 and AM30 were studied.

Figure 1 shows the microstructure of AZ31 in directions parallel and transverse to the rolling direction. (fine-grain structures are seen in the skin-layer.)

II-2. electrochemical property: Electrochemical tests were conducted to study the effects of anisotropy on corrosion. Wrought Mg alloys (made by rolling or extrusion) are known for their anisotropic mechanical properties. Their tensile and fatigue properties are well studied. However, the anisotropic corrosion property is not as well documented and it can be an important factor in the stress corrosion cracking susceptibility of these alloys. Figure 2 shows the electrochemical polarization behavior of AM30 when tested along its longitudinal and transverse direction.

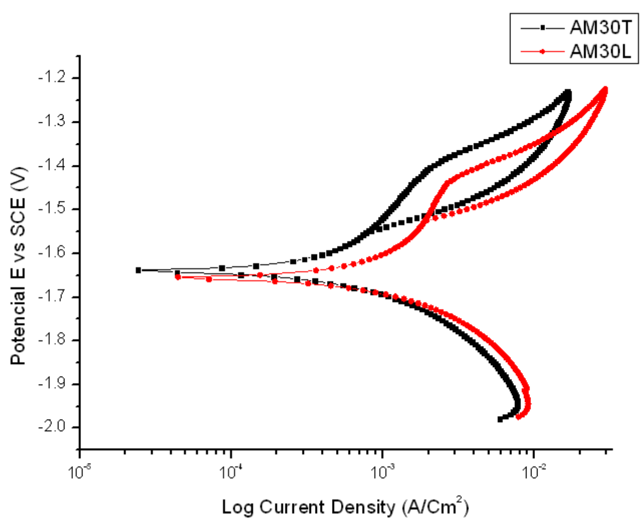


Figure 2. Polarization curves of AM30 measured along transverse and longitudinal directions. (GM9045P Solution: 0.9 wt.% NaCl + 0.1 wt.% CaCl + 0.25 wt.% NaHCO3, at room temperature.)

II.2 Mg coatings: Three types of Mg coatings were studied in this task. They are 1) commercial benchmark Alodine5200 (conversion coating) 2) an anodizing and 3) Al-based powder coating on Mg produced by cold-spraying. Examples of Mg-coating and the coating-interface studied are provided below.

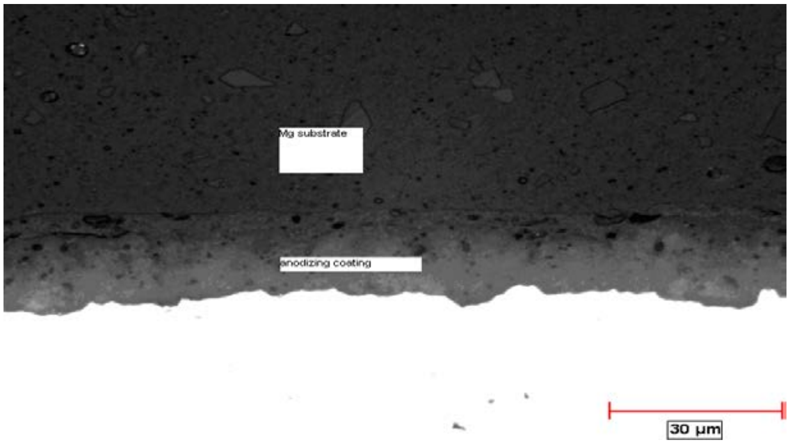
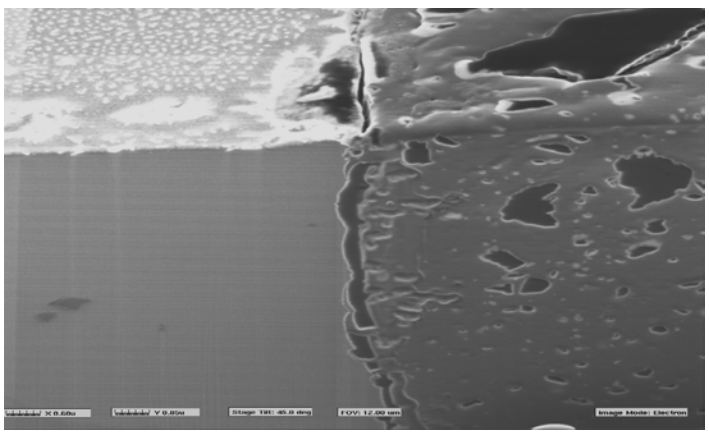


Fig 3. Cross-section of an anodizing coating showing a thick coherent anodizing layer (~20 microns thickness) on Mg.



(Mg alloy) (Alodine layer) Powder-coat

Figure 4. FIB/SEM image of the interface of powder-coating and Mg substrate, with a thin (<0.3 micron) Alodine5200 pre-treatment layer in between.

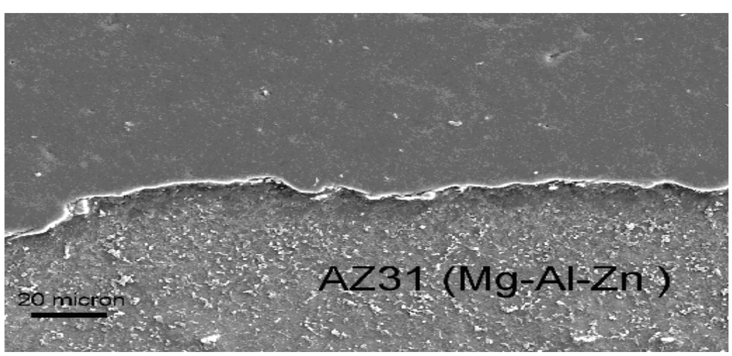


Figure 5. SEM image of the interface of Mg alloy and Al-based metal powder coating produced by cold-spraying.

III.3 SCC tests on cold-sprayed AZ3: Al-based coating and a mixture of Al and Mg, Al(50%)-Mg(50%), were applied on Mg AZ31 plate and SCC test samples.

Al alloys are generally more compatible with Mg alloy, in terms of their electrochemical potential versus that of Mg alloys. However, when pure Al or commercial Al alloys such as 6061 or 5052 still have some electrochemical driving forces on Mg corrosion when they are coupled with Mg material. Therefore part of the cold-spray work is to design metal powder mixtures or alloys that can best match the electrochemical characteristics of the underlying Mg substrate.

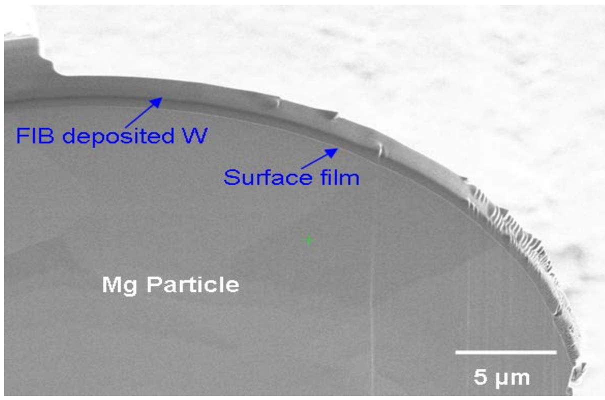


Figure 6. Morphology of thin (sub-micron) oxide on Mg powder, as studied in a related MTL project on metal powders. (Courtesy of Jian Li, NRCan-MTL).

It was found that the Mg powders can be very prone to surface oxide formation during the powder-making process. Figure 6 shows an example of such oxide on Mg powder surface. The influence of the powder quality and especially contaminations in the powders is being investigated.

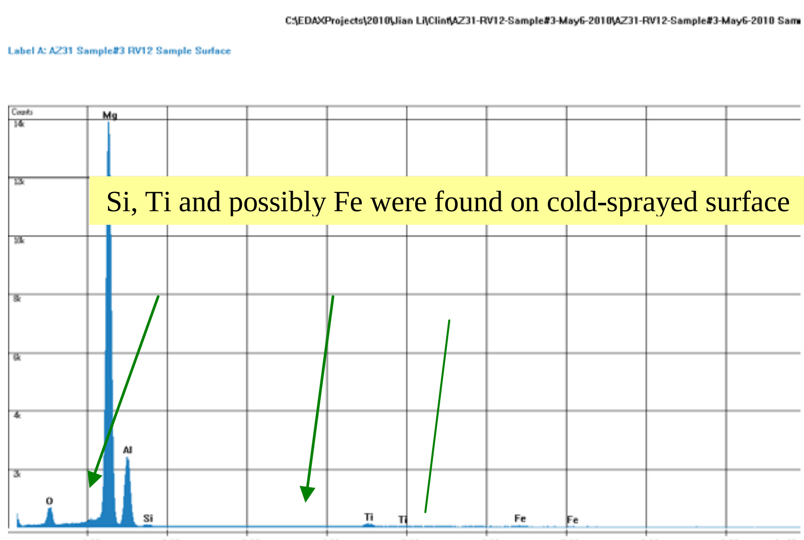
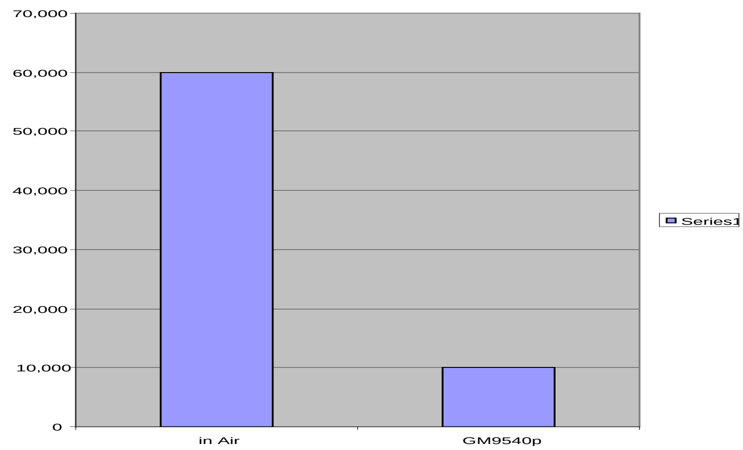


Figure 7. Presence of contaminants on the surface of AZ31cold-sprayed with alumina powder.

The other important finding during the cold-spraying study was that impurities can be incorporated onto the surface of Mg alloys if the cold-spray nozzle system has been used for spraying other metal/alloy powders. The residual elements left in the nozzle system can have a significant detrimental effect of Mg corrosion as this metal is very prone to micro-galvanic corrosion in the presence of more noble elements.



(90% of yield, R=0.90, F=1.0Hz)

Figure 8. The effect of GM9540 solution (0.9 wt. % NaCl + 0.1 wt. % CaCl + 0.25 wt. % NaHCO3) on the fatigue property of AZ31 alloy.

Even with a cold-sprayed coating, AZ31 is still prone to environmental degradation when immersed in a GM9540P solution. Figure 8 shows the reduction in fatigue life when the cold-sprayed Az31 is immersed in GM9540P solution.

<u>III. Other highlights of 2009 included:</u>

(1) Completion of a book chapter on protection of Mg alloys by cold-spray (an INTECH publication).
(2) Completion of an annual report on Mg corrosion for the Magnesium Front End Research and Development project (MFERD Task 1.4).
(3) Meetings with USA and China Mg corrosion participants in Canada and in USA.

5. Exchange of Information during the reporting period:

In the past year, two (2) presentations were made at the China-USA-Canada joint Mg workshop. (July 2010 teleconference meeting) and October 2010 in Ann Arbor, MI.

In additions, two (2) presentations on Annex V were made at IEA-AMT meetings (June 2010, Shanghai and Nov. 2010 in ORNL)

6. Technical Objectives: Is the project meeting the technical objectives as outlined in the Annex plan?

Yes. The project meets the time lines of the Annex V as contained in the operating plan.

7. Changes to Annex scope or objective:

There is no change in the scope and objectives of Annex V.

8. Publications:

1. A book chapter entitled 'Corrosion Protection of Magnesium Alloys by Cold Spray' has been accepted for publication by INTECH. A revised version is being processed by the publisher.

2. Annual Task 1.4 report for MFERD. Pp151-158. Pub. Oct. 2010. (A MFERD special publication)

INTERNATIONAL ENERGY AGENCY
CO-OPERATIVE PROGRAMME ON ADVANCED MATERIALS FOR TRANSPORTATION APPLICATIONS (IEA-AMT)

Annex VI: ***Co-operative Programme Low Cost Carbon Fibre Composites for Transportation Applications***

Covering the period: Jan 1, 2010 to Dec. 31, 2010

1. Title of Annex: Co-operative program on low cost carbon fibre composites for transportation applications

2. Annex participants:

UK Alan Wheatley (UK), University of Sunderland (UK)
USA Dave Warren, ORNL (USA)

3. Annex Objectives:

The annex objectives are to promote cooperation and information sharing between the participants and where appropriate, jointly develop test methods and standards that are crucial to the development of light weight materials for transportation applications.

3.1 Subtask I. Technical Information Exchange

The objective of this task is to achieve a balanced exchange of information among participants, relating to the testing and characterization of carbon fibre composite materials. Non-commercial, non-sensitive information/data will be shared. Such information might include materials characterization data collected by round-robin testing, the nature of test methods/techniques/equipment employed, and information on issues concerning test compatibility (See also Sections 2.2 and 2.3). Information exchange need not be limited to immediate Annex VI collaborators. All AMT participants could benefit from such information sharing (and vice versa). Activities will include:

- Exchange of reports, studies, and test samples as agreed upon in advance by the participating members on a semi-annual basis.
- Joint technical workshops and meetings.
- Reciprocal invitations to workshops and conferences.
- Reciprocal visits to research facilities.

Deliverables

The outline above covers the nature and scope of the information exchange process itself. In addition, the following specific deliverables are proposed.

3.1.1. Agreement on nature, scope and detailed content of Annex VI (Dec 2009).

3.1.2. Continue US/UK liaison at government department level (e.g. US DOE / UK TSB) to secure details of alignment of strategies on low cost carbon fibre composites for automotive applications (and beyond) (Dec 2009).

3.1.3. Reciprocal US/UK visits to consolidate established research links – (ongoing/perennial, 2010-2012).

3.1.4. Secure further national collaborators in this Annex to ensure international impact (2010 - 2011).

3.1.5. Share information on the basis of future agreements on the scope and nature of testing/characterization/standardization as outlined in Sections 2.2 and 2.3 - (perennial, 2010-2012).

3.2 Subtask II. Characterisation of Carbon Fibre Composites

There are a myriad of materials variables which play an important role in determining the properties and behavior of polymer-matrix carbon fibre composites. Relating materials variables to resulting properties is an essential component of this research. While some of this type of work will be subject to commercial restrictions, significant benefit could still accrue from collaborative efforts into materials characterization and information exchange between collaborators (e.g. for subsequent modeling or simulation).

Characterization tasks may include:

- Mechanical properties of carbon fibers composites
- Mechanical properties of polymer matrix materials.
- Mechanical properties of CFRP materials of various fibre/resin configurations (e.g. thermoplastic -v- thermosetting resins, laminated -v- non-laminated composites, continuous -v- discontinuous fibers, fibre content and orientation).
- Thermal analysis / cure kinetics of thermosetting resin systems.
- Interfacial characterization.
- Rheological characterization of resin systems and thermoplastic composites.

Deliverables

A full characterization effort covering all of the above fields is not possible within the scope of AMT. Furthermore, much characterization work will be ongoing outside of AMT. Hence the following short-medium term deliverables are proposed:

3.2.1. Agreement on precise detail of the characterization effort to be conducted within the definitions outlined in 2.2 above (2010).
3.2.2. Work timetable drawn up following Step 1 above and disseminated to stakeholders in advance of the next AMT Exco (2010).
3.2.3. Information relating to this work shared via the mechanisms outlined in Section 2.1 above (perennial, 2010-2012).
3.2.4. Qualitative and quantitative evaluation of the impact of information sharing. Such evaluation results to be shared with stakeholders (perennial, 2010-2012).

3.3 Subtask III. Design Data and Standardisation

With the benefit of data such as that generated in 2.2 above, mathematical models and simulations can be developed to help predict the behavior of CFRP materials in automotive applications. Such models may be related to:

- Static mechanical behavior
- Dynamic mechanical behavior
- Impact behavior
- Creep behavior
- Failure analysis
- Flow behavior during processing
- Cure kinetics
- Interfacial phenomena

The combination of comprehensive characterization data and reliable predictive modeling tools will result in a bank of design data – thereby helping to alleviate one of the constraints on CFRP adoption, namely lack of confidence or comfort on behalf of the automotive industry in its adoption.

Reliable materials data and predictive modeling tools are vital to engender confidence in the material. In a similar manner, standard uniform test procedures must be employed to generate such design data – otherwise diverse sets of data will not be directly comparable. For novel materials such as low cost CFRP, the development and acceptance of standard test techniques are a vital step in its adoption. The aim of this task is therefore to formulate a set of relevant test methods and achieve consensus on the formulation.

- Establish a database of various relevant test methods currently used for evaluating carbon fibre composites.
- Liaise with industrial and government stakeholders (e.g. standards institutions) to identify any critical "gaps" in test standard provision which currently constrain development and/or adoption.
- Develop relevant test techniques and standards via international round robin testing, evaluation and information exchange.

Deliverables

3.3.1. Agreement on precise detail of the design data and standardization effort to be conducted within the definitions outlined in 2.3 above (2010).

3.3.2. Work timetable drawn up following Step 1 above and disseminated to stakeholders in advance of the next AMT Exco (2010).

3.3.3. Information relating to this work shared via the mechanisms outlined in Section 2.1 above (perennial 2010-2012).

3.3.4. Qualitative and quantitative evaluation of the impact of information sharing. Such evaluation results to be shared with stakeholders (perennial, 2010-2012).

4. Technical Progress and Accomplishments for the Reporting Period:

For CFRP to contribute to major weight and fuel savings in the automotive sector then research is required into all aspects of low cost carbon fibre development (e.g. precursor choice, processing, surface treatment etc). However, research in many aspects such as selection of precursor materials and associated manufacturing processes is commercial and/or sensitive in nature. As such it is not compatible with the open information-sharing philosophy of IEA IA-AMT. Hence, such research is now being undertaken outside of Annex VI's remit. For this reason, Annex VI was extensively revised over the previous reporting period and much of the progress to date had been in achieving consensus on a new viable Annex VI programme definition. The results of this extensive modification can be seen in the Annex VI objectives and deliverables outlined in Section 3 of this report.

In summary, Annex VI now concentrates on information-sharing and consensus-building in those aspects of low cost carbon fibre research which are outside of the competitive arena (pre-competitive, non-commercial and/or non-sensitive in nature).

In terms of progress against these new objectives, a major development is now underway which aims to identify all relevant standards against which carbon fibre performance is currently assessed and to build a consensus on the suitability of these standards in both range and content. This study involves the annex contributors listed in Section 2, together with major stakeholders from industry and academia. The list of contributors to this work is growing significantly as the work progresses. In support of this aim, an industrial forum was convened at the recent "***Global Outlook for Carbon Fibre***" Conference in Valencia, Spain in September 2010. This session was chaired by a panel of 4 people; the author (AW), Dave Warren (DW) of ORNL, Andy Foley (AF) of SGL Carbon Fibers and Roland Thevenin (RT) of Airbus. AW gave an overview of the work and, in particular, its IEA context. DW presented a summary of the low cost carbon fibre programme underway at ORNL and introduced the subject of development and harmonization of standards relating to carbon fibre. AF and RT gave industrial perspectives from the point of view of fibre producers and end-users respectively. Feedback (i.e. on the need for standard development and harmonization) was sought via questionnaire from the delegates and this is currently being analyzed for presentation to the AMT ExCo. Following the event, it has been agreed to set up and formalize a working party/steering group incorporating senior stakeholders to drive this process forward. This is ongoing.

The significance and impact of this work is outlined in Section 3.3.

5. Exchange of Information during the Reporting Period:

Information exchange during the reporting period took place at:

5.1 AMT ExCo meeting, Shanghai, June 2010.

5.2 "***Global Outlook for Carbon Fibre***" Conference in Valencia, Spain, 29-30 September 2010. Alan Wheatley and Dave Warren attended and presented material on Annex VI (see Section 4 above).

6. Technical Objectives:

Annex VI was completely revised towards the end of the previous reporting period and the technical objectives defined as per Section 3 are being pursued. Current progress against these technical objectives is on target. Current activity suggests great potential for the newly-defined annex and its objectives.

7. Changes to Annex Scope or Objective:

Annex VI was completely revised over the previous reporting period and these revisions were endorsed unanimously by the AMT Executive Committee. No further changes have been introduced or proposed during the current reporting period.

INTERNATIONAL ENERGY AGENCY
CO-OPERATIVE PROGRAMME ON ADVANCED MATERIALS FOR TRANSPORTATION APPLICATIONS (IEA-AMT)

Annex VII. Co-operative program on development, evaluation and standardization of methods for testing mechanical properties of nanomaterials for application in automotive industries

Covering the period: Jan 1, 2010 to Dec. 31, 2010

1. Title of Annex: co-operative program on development, evaluation and standardization of methods for testing mechanical properties of nanomaterials for application in automotive industries

2. Annex Participants:

Germany:	Dr. Michael Griepentrog, Lead investigator, BAM, Germany
USA:	Dr. Stephen Hsu, GWU, USA
	Dr. Chanmin Su, VEECO, USA
	Dr. Gregory F. Meyers, The Dow Chemical Company, USA
China:	Dr. Guanglu, National Center for Nanoscience and Technology,
	Dr. Chen, Shanghai Institute of Ceramics, CAS, China (new)
Canada:	Michel Dumolin, Materials and Processes, Canada (new)
	Florence Perrin-Sarazin, Institute Industrial Materials, NRC,
UK:	Dr Alan Brewin, NPL, UK

3. Annex Objectives:
The general objective of this Annex is to conduct cooperative research on development, evaluation and standardization of methods for testing mechanical properties of nanomaterials for application in automotive industries and to establish the nanoscale measurement methodology that correlates with the measured global property.

Nanomaterials have great potential for the topics in transportation technologies (e.g. polymer/clay nanocomposites for light-weighting, polymeric thin films for electronics, and friction-reduction/control interfaces). Standard nanomechanical property test methods for this class of materials do exist, however, the complexity of wide range of nanomaterials, their composite combinations, and accuracy and precision of measuring the influence of nanoparticles on the global properties are still in flux. This annex aims to examine the two most commonly used instrumentations: nanoindenters and atomic force microscopes for their efficacy in measuring the nanomechanical property of a nanoparticle in a matrix as well as to compare the results on the same material.

4. Technical Progress during the reporting period:
Following activities were planned for the first year:

Technical Information Exchange.
Definition and global characterization of the main materials under investigation.
Definition of the preparation procedure.

Development and evaluation of the round robin design.
Evaluation and standardization of IIT test methods for composites.

Following steps in preparation the planned round robin have been done:

Sample preparation and conditioning:

Further preparation techniques like microtome cutting have been tested. As result of these tests BAM will buy a microtome and all samples for investigation in the round robin will be prepared by cutting with this new instrument. The samples will be ready for delivering towards the end of 2010.

Development and evaluation of the round robin design.

The design of the round robin testing using nanoindentation and AFM techniques was completed and successful tested by the partners BAM, Germany and GWU, USA.
To finalize the experimental procedure for the planned round robin the first discussions with the annex partners have taken place.

Evaluation and standardization of Instrumented Indentation Test (IIT) test methods for composites

During the last meeting of ISO TC 164 SC 3 (Leader of Annex VII Dr. Michael Griepentrog, Germany was the acting chair of this meeting) following resolutions, important for Annex 7 have been adopted:

To start the work on standardization of indentation testing on polymers.
Resolution N° 216 *SC 3 agreed to maintain the Liaisons with ISO/TC 17/SC 20 and ISO/TC 61/SC 2.*

To start the work on standardization on dynamic indentation testing.
Resolution N° 224
SC 3 mandated the ad-hoc Working Group for the revision of standards series ISO 14577 to prepare a scope and a proposal for integrating dynamic instrumented indentation testing methods (including indentation creep and nano impact testing) into ISO14577 in advance of the 2011 meeting of SC 3. This could include preparation of a NWIP for a 5th Part of ISO 14577.

Both resolutions will be taken in to account for the future of Annex VII.

AFM test method development

AFM has the necessary small tip size to probe nanoparticles below 100nm diameter but lacks the stiffness of the nanoindenters in measuring the indentation marks precisely produced when the tip impressed on the surface of the nanoparticle. At the same time, the nanoindenters use natural diamond tips for its hardness and stiffness but it is difficult to fabricate tip diameter smaller than 100nm. Many of the nanoparticles used in making composites are smaller than 50nm, thus making measuring nanomechanical property of a single nanoparticle using a much bigger tip very difficult.

Working with Veeco Instrument (now Bruker), new measurement techniques have been developed to overcome many of the issues, such as using stiff cantilevers, blunt tips to avoid penetration into the surface, and ultra-sensitive imaging techniques to accurately measure nanometer scale impressions. Veeco has committed in supplying necessary software and hardware for AMT study.

GWU has drafted a preliminary AFM test procedure for participants to use for such measurements.

5. Exchange of Information during the reporting period:
Presentation of the main goals of the round robin and the first draft of the round robin on the ExCo meeting June 2010 Shanghai.

Exchange information of the planned Annex VII activities with the Chairman of VAMAS TWA 22 Dr. Nigel Jennet UK and the Chairman of VAMAS TWA 33 Prof. Leszek Utracki, Canada and to learn their activities in similar activities.

6. Schedule: Is the project on schedule?
Yes.

7. Technical Objectives: Is the project meeting the technical objectives as outlined in the Annex plan?
Yes
A first project meeting is planned but the date is not yet fixed.

8. Changes to Annex Plan:
No changes to the Annex.

9. Publications:
"Measurement of nano- micro-particles in a polymer matrix using AFM," Price, W. and Hsu, S.M., Proceedings of the 12th International Congress on Mesomechanics, National Taiwan University of Science and Technology (NTUST) in Taipei, Taiwan, June 21 to 25, 2010.

INTERNATIONAL ENERGY AGENCY
CO-OPERATIVE PROGRAMME ON ADVANCED MATERIALS FOR TRANSPORTATION APPLICATIONS (IEA-AMT)

Annex VIII Thermoelectric Materials for Waste Heat Recovery: An International Collaboration for Transportation Applications

Covering the period: Jan 1, 2010 to Dec. 31, 2010

1. **Title of Annex:** Thermoelectric Materials for Waste Heat Recovery

2. **Annex Participants**:

USA:	Dr. Hsin Wang, ORNL, USA
	Prof. Terry Tritt, Clemson University, USA
	Dr. Alex Mayolett, Corning Inc., USA
	Dr. Fred Harris, ZT-Plus Inc. USA
	Dr. Jeff Sharp, Marlow Industries USA
Canada:	Dr. Jason Lo, CANMET, Canada
	Professor Holger Keinke, University of Waterloo, Canada
China:	Professor Lidong Chen, Shanghai Institute of Ceramics, CAS, China
Germany:	Dr. Harold Bottner, Fraunhofer Institute for Phys. Meas, Germany

3. **Annex Objectives**:

The annex aims to develop test methods for the evaluation of thermoelectric materials and to develop precision statements for the standardization of these methods.

Specifically, we aim to:

- Develop standard testing methods and procedures for thermoelectrics (bulk and nano-composite materials)
- Assess the state-of-the-art for thermoelectric materials and identify critical issue to improve performance
- Conduct international round-robin tests of standard thermoelectric materials (20-500ºC)
- Exchange Technical information
- Characterize key properties (transport properties and others using advanced tools such as STEM and neutron scattering) of potential thermoelectrics for transportation applications

4. **Technical Progress and accomplishments for the reporting period**:

The first international Round-robin study:
Thermoelectric materials were purchased from Marlow Industries. Both n-type and p-type Bi_2Te_3 specimens were machined to the specifications agreed by annex participants. The first set of round robin specimens were sent out to participating labs in early 2010. Each lab was given 8 specimens to conduct thermal diffusivity, specific heat and electric conductivity and Seebeck

coefficient measurements. The labs were instructed to use their common practice to measure these samples. The results were gathered and discussed at the annex meeting held in conjunction with the International Thermoelectric Conference in Shanghai in May 2010. We discovered major problems in specific heat and electrical conductivity measurements. Some of the results are shown in Figures 1-4. The efforts by annex participants were highly appreciated by the conference organizers and attendees.

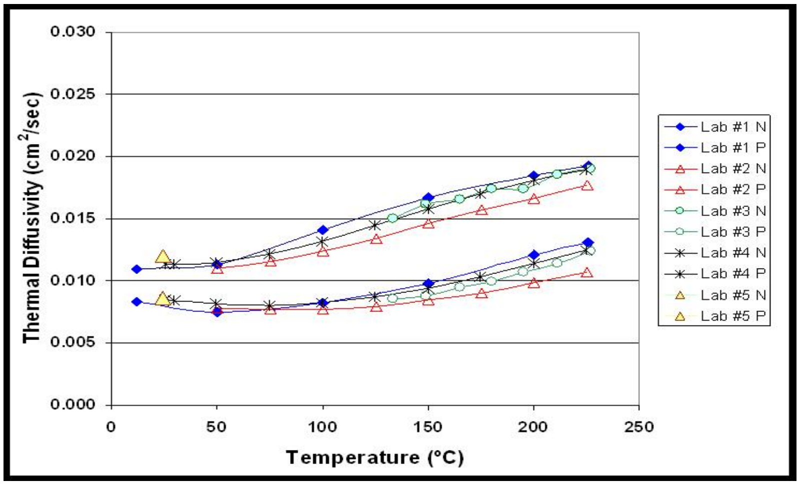


Figure 1. Thermal diffusivity measurements of p-type and n-type bismuth telluride

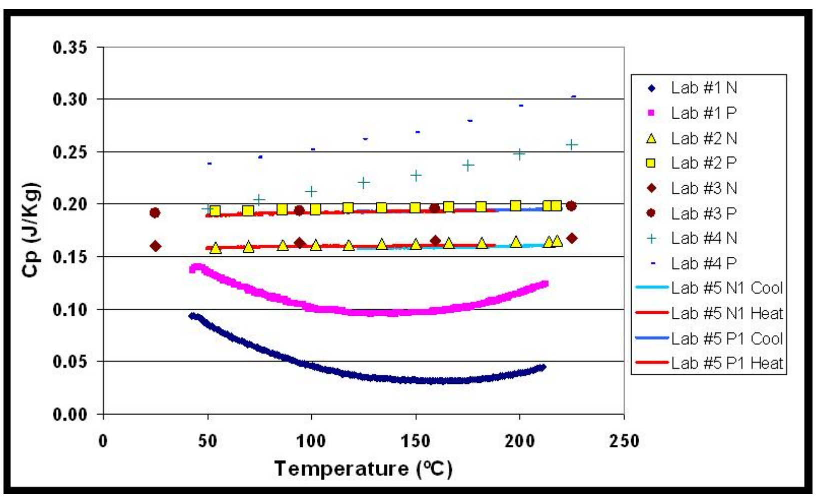


Figure 2. Specific heat results showed major issues in DSC measurements

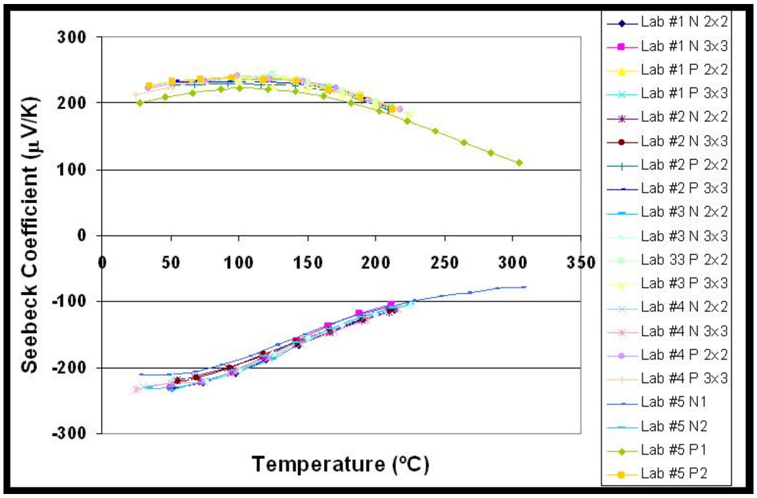


Figure 3. Seebeck coefficient results showing good agreements among labs

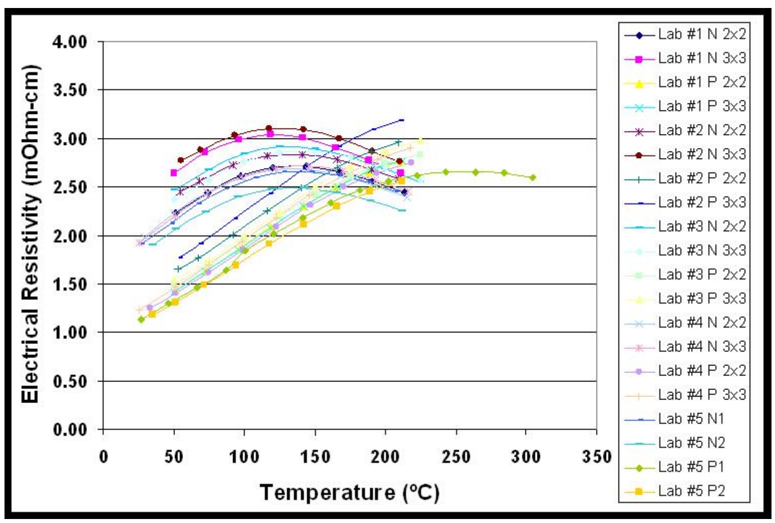


Figure 4. Electric resistivity measurements showed significant differences among labs

Second international Round-robin study:

After the discussion on the first round-robin results, a decision was made to start the 2nd round robin test focusing on the p-type Bi_2Te_3 materials. The reason was that the p-type material showed better sample-to-sample consistency. Instead of testing different sets of samples, two sets of p-type specimens were sent among participating labs in September 2010. The tests are scheduled be completed by the end of 2010.

The main objective of the round-robin tests is to assess the state-of-the-art of existing measurement technologies. Based on this, test procedure will be tightened to establish a standard test procedure for the determination of ZT.

5. Exchange of information during the reporting period:

Two presentations during the Exco meetings in May and November, 2010. Annex meeting was held in Shanghai in May 2010.

6. Technical Objectives:
No change in the technical objectives for the annex. Since June 2009, the technical activities have been planned and systematically implemented.

7. Changes to annex scope or objective:
None

8. Publications:
None