

# AMPERE Newsletter

# **Trends in RF and Microwave Heating**

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# President's message



Cristina Leonelli President; AMPERE EUROPE Contact Email: cristina.leonelli@unimore.it

Dear AMPERE members and friends,

My sincere apologies for having closed the Association website (www.ampereeurope.org) for such a long time, however, I hope that you will be satisfied with its new look.

Firstly, the European Union General Data Protection Regulation (GDPR) has required a really significant modification to the old existing website, so we took the opportunity while we made the necessary steps to ensure that the website could be accessed via portable devices. Additionally, we have incorporated the Newsletter website within the Association website, offering a single and compact information source.

As you will appreciate, the AMPERE website has restored all the previous member services, but we have also instituted new ones, such as the "search" operation in the Newsletter website and the links to the confederated Associations.

I would hope that those of you who have any information about events and approved projects, will readily be willing to prepare a short paragraph for the News, so that to ensure a more active and vivid website. The introduction of a new page fully dedicated to the events organized and endorsed by AMPERE provides a data bank for past and future events which can be enriched with new endorsed events.

The cost of the website restyling has been just slightly higher than the usual 2-year maintenance cost which effectively reduces the Association's balance as approved during the September 2017 OGA in Delft. I conclude this letter listing the three events already planned for the year 2019:

**HES-19**, Heating by Electromagnetic Sources, 22-24 May 2019, Padua (Italy), including a **UIE Ph.D. Intensive Course**.

5thSummerSchoolinHighEnergyProcessing,Ultrasound&MicrowaveTechnologies,26-28June2019,Faculty ofChemicalEngineeringandTechnology,CracowUniversity ofTechnology (Poland).ValueValue

**AMPERE 2019**, 17th International Conference on Microwave and High Frequency Heating, 9-12 September 2019, CPI, Universitat Politècnica de València, Spain.

I hope that we may soon receive new links of our members' activities, which will be added in alphabetical order in the topic: "About us".

I will be pleased to receive as much feedback as possible on the new website, particularly from those groups that have been granted industrial projects and desire to promote their new websites.

Best regards,

Cristina Leonelli President, AMPERE EUROPE.

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# Dry Microwave-Chemistry Enabled Fabrication of Pristine Holey Graphene Nanoplatelets with Rich Zigzag Edges <sup>#</sup>

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<sup>#</sup> A paper presented at IMPI 52, Long Beach, CA, USA, June 26-28, 2018.

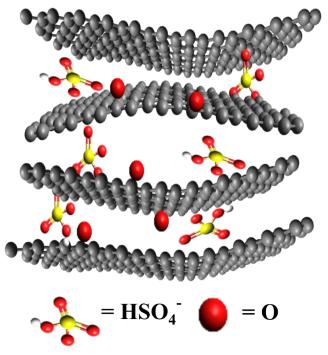
#### 1 Introduction:

Holey graphene, referred to as graphene with nanoholes in their basal planes, recently attracted increasing research interests from both fundamental and practical application point of view [1]. The existence of nanoholes in these bulk 3D materials not only increases accessible surface area, but also provides desired "short-cuts" for efficient mass transport across graphene basal planes and ultimate access to, and from inner surfaces, which is very different from the intrinsic perfect graphene sheets. Most importantly, generating nanoholes naturally transform a large number of in-plane atoms into edge atoms. These atoms have different electronic states and chemical functionalities from their basal planes, which render holey graphene materials unique properties and capabilities.

Recent years have witnessed wide range of applications from molecular sensing to electrochemical energy generation and storage, demonstrating the critical advantages of holey graphene-based materials toward practical applications. However current approaches for scalable production of holey graphene materials require graphene oxide (GO) or reduced GO (rGO) as starting materials [2]. Thus generated holey graphene materials still contain a large number of defects on their basal planes, largely due to the existence of or evolved to "hard-to-remove" defects in these starting materials. The existence of these defects not only complicates fundamental studies, but also influences certain practical applications due to the largely decreased conductivity, thermal and chemical stability.

#### 2 Methodology:

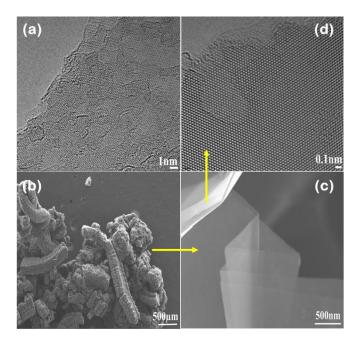
This work reports a novel, rapid, and eco-friendly approach for mass production of holey graphene materials [3]. In this approach, dry microwave chemistry was exploited to fundamentally solve the above mentioned problems, so that controllable generation of nanoholes can be achieved while leaving other parts of the graphene basal plane largely intact. Furthermore, the hole edges can be controlled to be rich in zigzag geometry, which is the preferred edge structure for catalytic and spintronic applications.

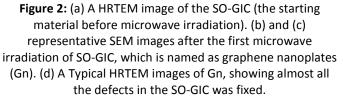


**Figure 1:** A schematic drawing to demonstrate the structure of the slightly oxidized GIC (SO-GIC). Low density of oxygen containing groups were introduced in the basal planes during oxidation and some intercalants (HSO<sub>4</sub><sup>-</sup>) still exist in the galleries between the graphene sheets.

One of the key features in our new approach is the use of slightly oxidized graphite intercalation compounds (SO-GICs) (Figure 1 and Figure 2a) instead of direct use of GO or rGO as starting materials. In SO-GICs, a low density of oxygencontaining groups are sparsely distributed across the graphene sheets in entire graphite flakes. Hence, the formation of those hard-to-remove defects as described during annealing of GO/rGO would be largely prevented.

The second key feature of this approach is to make full use of microwave heating in air instead of traditional convection heating in inert environments (such as in Ar). For carbon materials with only slight oxidation, strong microwave absorption and rapid transformation of the absorbed MW energy into heat with high efficiency are expected [4-6]. The heat generated can achieve a very fast temperature rise, which can induce rapid chemical reactions. In this case, the rapid chemical reactions induce fast degassing, building up an inner gas pressure inside SO-GICs, which pushes the graphene sheets apart (expansion). Furthermore, the mechanism of microwave heating of slightly oxidized carbon materials without solvent is mainly wireless (contactless) Joule heating [5-6]. The electromagnetic field of microwaves induces motion of electrons in conducting material, which causes heating due to electrical resistance [5-7]. Therefore, the current induced during microwave irradiation is not converted to heat in perfect graphene domains due to their ballistic conduction behaviour. On the other hand, the defective regions, in which oxygen containing groups or other topological defects are located, scatter the electrons and provide electrical resistance. Therefore, these defect regions are headed selectively. This region-selective heating can induce different chemical reactions depending on the microwave power, irradiation periods, and the chemical nature of carbon materials subjected to microwave irradiation. These reactions may include (1) direct carbon combustion selectively at the defective regions to generate vacancies or nanoholes (direct perforation); and (2) deoxygenation and reconstruction of the defect sp<sup>3</sup> carbon bonds into sp<sup>2</sup> configuration (fixing defects or remediation). These two processes tend to occur concomitantly and competitively. With this consideration, microwave irradiation pattern was optimized to generate graphene materials with controlled hole structures. Most importantly, all the graphene materials generated by this approach have their basal planes (in the non-hole regions) nearly free of other defects. This feature ensures the preservation of the outstanding electric and thermal transport properties, and chemical inertness of pristine graphene.

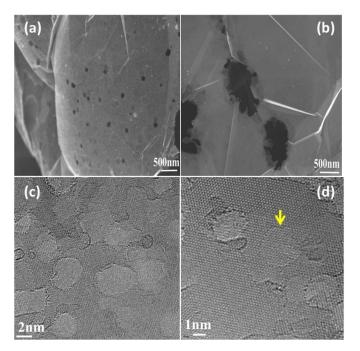




#### 3 Results and Discussion

We first applied lower power and shorter microwave irradiation time to exfoliate (or expand) the SO-GICs (Figure 2a-b). The product of this step was named as graphene nanoplatelets (Gn) due to the fact that most of the defect in the basal planes were concurrently fixed during the expansion and most of them composed by several layers of graphene (Figure 2cd). The efficient expansion of the SO-GICs warranted most of the graphene sheets in the resulting Gn to be accessible to the oxidant (air) for direct combustion in the following step. Upon cooling down (~ 10 mins), the Gn was irradiated for another 5 seconds at 200 W. Further expansion was observed from Scanning Electron Microscope (SEM) imaging. It was also observed that holes of 300-500 nm were generated on the basal planes of most of the Gn. We referred to this sample as graphene nanoplatelets with holes (Gn-H), Figure 3a). Since both sides of the exfoliated nanoplatelets in Exp-Gn were accessible to the oxidant (air), holes were generated on both sides of the nanoplatelets. Upon one more 5-second microwave irradiation (upon cooling down, ~ 10 mins), the surface area of the product was further increased to 744  $m^2/g$ . The holes became larger and most of the holes penetrated deep into the lower layers of the stacked graphene sheets. Some of them even penetrated through the entire nanoplatelets. Note that most of the chemical etching approaches struggle to create holes which penetrate through a nanoplatelet of several layers due to the preferred in-place etching processes [8]. Furthermore, the hole edges are heavily etched, rough and irregular (Figure 2b). We termed this sample as graphene nanoplatelets with etched hole edges (Gn-HE). Very interestingly, in the non-hole regions from SEM, baby holes with diameters of 2-5 nm were observed via high resolution Transmission Electron Microscope (TEM) (Figure 2 c-d). Some of the baby holes were terminated with zigzag edges (Figure 3d). The near pristine nature of their basal planes and the richness of the zigzag edges were clearly observed via atomic resolution TEM and further supported by other techniques, including the studies of the localized  $\pi$ -edge states associated with zigzag geometry via electron paramagnetic resonance (EPR) measurements.

The holey graphene nanoplatelets were explored as metal-free, even heteroatom-doping free- chemical catalysts for hydrogen atom transfer reactions. They exhibited excellent catalytic activity, desired selectivity, and chemical stability for recyclability, which were not achievable by their counterpart holey graphene derivatives with basal plane defects. Equally important, the intrinsic chemical catalytic activity of localized  $\pi$ -edge states was unambiguously demonstrated experimentally in this work, due to the absence of other defects in the basal planes of these unique holey graphene nanoplatelets. Recently, the electrochemical catalytic behaviour of the holey graphene nanoplatelets was also studied and carefully compared with a holey graphene materials fabricated from GO/rGO, which contained a large amount of other defects. Using oxygen reduction reaction as model system (ORR), for the first time, we found the catalytic current density is proportional to the concentration of the paramagnetic species measured by EPR, unambiguously suggesting that the zigzag edges in holey graphene materials are the catalytic sites for ORR. Furthermore, even though the amount of catalytic sites in our holey graphene nanoplatelets is much less than those in the holey graphene materials fabricated from GO/rGO, the catalytic efficiency of each catalytic site is much higher, which is comparable to those metal-based catalytic sites.



**Figure 3:** (a) and (b) A representative SEM image of Gn-H and Gn-HE respectively. (c) and (d) Typical HRTEM images of SOG-HE, showing baby holes of 2-5 nm and nearly defect free honeycomb structures of the basal planes. The edges of some of the baby holes were in zigzag geometry, indicated by a yellow arrow.

It is worthwhile to mention that there are no scalable approaches reported to date for mass production of highly conductive and chemically stable pristine holey graphene without involving metal-containing compounds and at low cost for practical bulk applications. Furthermore, the traditional methods are challenging in controlling the hole edges with dominated zigzag geometry. From thermodynamic point of view, armchair should be the major ones. We attribute the unexpected results presented in this work to the unique contactless heating mechanism provided by microwave heating applied in this approach compared to traditional convection heating approaches and also the structure of the slightly oxidized graphite as the starting materials for the microwave chemistry.

#### 4 Summary and Conclusions

In summary this work reports a simple and scalable approach to fabricate pristine holey graphene nanoplatelets which are rich in zigzag edges. The approach exploits dry microwave chemistry providing several non-competitive advantages over the previously reported methods for large scale production and broad application. (1) The structures of graphene products (with or without holes in their basal planes) can be controlled to give materials that are nearly free of other defects in their basal planes. This feature ensures the preservation of the outstanding electric and thermal transport properties, and chemical inertness of the basal plane of graphene; (2) The process is eco-friendly, no metalcontaining compounds are involved in the production process; (3) The microwave fabrication process is fast (several seconds). In addition, compared to the approach based on wet microwave chemistry [9], there are additional advantages brought in by this dry microwave chemistry approach: (a) The issues associated with the usage of strong acids and oxidants in a microwave oven, such as safety and corrosion to microwave instruments, are naturally avoided. (b) Since the dry approach is a solvent free process, all the microwave energy is efficiently absorbed only by the reactants. Accordingly, the required microwave energy is much lower and the irradiation time for the production is even shorter [9]. All these advantages greatly simplify the design for a continuous largescale and cost-effective production of high quality holey graphene nanoplatelets with controlled structures, which can bring a broad spectrum of potential applications of holey graphene materials to reality.

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#### About the authors

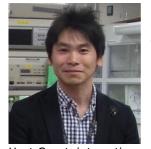


**Dr. Keerthi Savaram,** joined Rutgers, the State University of New Jersey at Newark, for the Master Program in Chemistry in 2011. Under the supervision of Prof. Huixin He, She obtained her PhD in Chemistry in 2017. Her PhD was focused on Microwave enabled fabrication of highly conductive graphene and porous carbon/metal

hybrids for sustainable catalysis and energy storage. Currently she is working as an analytical Chemist in R&D lab in Scientific Design Company, Inc. New Jersey, USA.



Qingdong (Jason) Li received a BSc in Chemistry from Jilin University, China in 2015. He is currently a majored Ph.D. candidate in Chemistry under the supervision of Dr. Huixin He. He is actively involved in the research of microwave enabled synthesis of nanomaterials for the application in electrocatalysis and energy storage.



Dr. Kazuyuki Takai is a full professor at the Department of Chemical Science and Technology of Hosei University, Tokyo, Japan. His research interests are in the area of Physical Chemistry of Condensed matter, in particular Electronic properties of Graphene and 2Dmaterial-based systems, and their Host-Guest interactions, including: Magnetic and Electron

transport properties, Chemical activities of  $\pi$ -electron systems such as Nanosized Graphene, Nanodiamond, and its controlling though the Chemical Modification; STM / STS and Raman spectroscopic characterization; design and fabrication of 2D Material-based Electronic and Energy devices. Dr. Kazuyuki TAKAI received his BS (1996) and PhD in Science (2001, Supervisor, Prof. Toshiaki Enoki) from the Tokyo Institute of Technology, Japan. In 2001, he joined the Department of Chemistry of Tokyo Institute of Technology as a research associate, and then as an assistant Professor. In 2013, he was promoted to an associate professor at the Department of Chemical Science and Technology of Hosei University, and became a full professor in 2016.



Dr. Vladimir Osipov received the degree of Electrical Engineer (Honors E.E.) in the field of "Optoelectronics and Optoelectronic Devices" in 1987 from Leningrad Electro-Technical Institute (LETI), Faculty of Electronics, Department of Optoelectronics. He joined the loffe

Physical-Technical Institute, Russia in 1987 as researcher and received his PhD on the specialty "Semiconductor and

Dielectric Physics" in 1994 from loffe Institute. He was appointed as Senior scientist in 1999. He was a visiting Associate Professor (2002) and JSPS Fellow in Tokyo Institute of Technology (2004-2005) and also occupied a position of JSPS Fellow in Hosei University (Tokyo) during 2017-2018. His research interests include magnetic, electron spin resonance and optical properties of edge-localized states in nanographites, defects in diamonds, and some issues of nonlinear optics. He is an expert in preparation of multishell nanographites and use the electron spin resonance technique for detecting  $\pi$ -electronic oxygen-sensitive edge-localized spin states of nanographene having unconventional electronic properties. Together with his international collaborators he first found the new unique X-band ESR signatures of NV<sup>-</sup> defect and multivacancy in nanodiamonds in the half magnetic field region. Currently he is the member of St.Petersburg team participating in the bilateral project under the Japan-Russia Research Cooperative Program. He is the author of more than 100 papers in refereed journals and books.



Dr. Huixin He is a full professor at the Department of Chemistry at Rutgers University, Newark NJ. Her current research interests include microwave chemistry to develop rapid and scalable approaches for various carbon and carbon hybrid nanomaterials. Her group explores the unique properties of these nanomaterials with innovative programmed assembly strategies to

integrate with other nanomaterials to achieve high performance applications in batteries, catalysts molecular sensors, and multifunctional drug delivery systems. Dr. Huixin He received her PhD (Supervisor, Prof. Zhongfan Liu) in Chemistry/Nanoscience from Peking University, China in 1997. She joined National University of Singapore as a research associate, working mainly on plastic microfluid channels and micropatterns by soft lithography. In 1999, she came to the United States and worked with Professor Nongjian Tao, at Florida International University and then moved to Arizona State University, on molecular electronics, including the electronic properties of metallic quantum wires and single chain conducting polymer wires. In 2002, she joined Chemistry Department, at Rutgers University in Newark.

# Glass-Like Functional Layers with Microwave-Generated Plasma-Enhanced Chemical Vapour Deposition (PECVD)

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#### 1 Introduction

Plasma is often referred to as the fourth state of aggregation. If energy is continuously supplied to a solid, the solid melts and then becomes a gas. Finally, the gas converts into a plasma, if the ionization energy of the gas particles is reached. Plasma thus consists of a gas or a gaseous mixture with a large number of neutral and charged particles in different excitation states. The lifetime of the charged particles in the plasma depends on the number of collisions between them. Low pressure conditions are therefore advantageous for many plasma processes in order to reduce the collision frequency. In addition, low-pressure processes prevent contamination, thus enabling high-quality layers to be deposited.

In plasma-assisted chemical vapour deposition (PECVD), the chemical reaction is supported by plasma. Using plasma, the surface temperature of the substrate can be significantly reduced and even temperature-sensitive materials can be coated without any thermal damage. The process uses gases, liquids or solids that can easily be evaporated under low pressure. These gaseous substances are introduced into the vacuum vessel, excited by the plasma, radicalized and deposited reactively on the substrate surface [1].

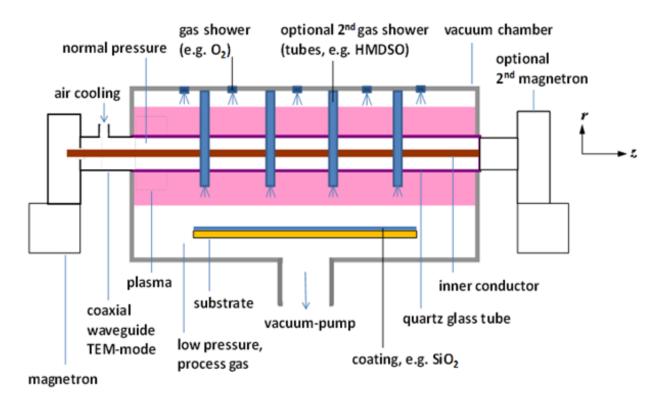


Figure1: Schematic sketch of a coaxial microwave plasma source (Plasmaline)

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PECVD processes differ in the energy source used to generate the plasma. If microwaves are used as an energy source, plasmas are generated with very low ion and high electron energy. Such plasmas are cold (<100 °C) and have a high reactivity, which enables a high coating rate of up to 20  $\mu$ m/min and more without surface damage. These plasmas are ideal for coating temperature-sensitive materials such as plastics.

This process is also economically competitive with established coating methods, because of very high coating rates, short process times and the possibility to coat very large surfaces instantly. Both compensate for the higher investment costs of the plasma system. The low-cost consumables silicone oil and oxygen additionally improve the economic efficiency. The coated areas do not have to be reworked [2,3]. Plasma systems essentially consist of an evacuated recipient, a vacuum pump, a plasma source and a gas inflow. The working pressure and gas composition are adjusted to requirement using the vacuum pump and the gas inflow. The plasma can be ignited and maintained by microwaves. By coaxially guiding the microwaves, linear plasma sources of several meters can be created, which ensure a homogeneous plasma density over their entire length (Fig. 1). If several sources are combined in parallel, the result is two-dimensional plasma sources that generate a homogeneous aerial plasma (Fig. 2).

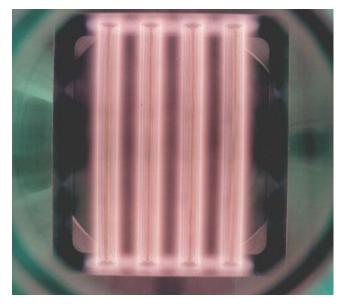


Figure 2: Aerial plasma array with burning plasma

The diversity of possible layers will be demonstrated using the precursor hexamethyldisiloxane (HMDSO) and oxygen as examples.

Under the influence of the plasma, the HMDSO precursor and the working gas oxygen, a transparent glass layer is deposited onto the substrate surface. If only the precursor HMDSO is exposed to a plasma, the monomers produced polymerize on the substrate to form a soft silicon-like layer. A glass layer or silicon-like layer can be continuously deposited during the process by varying the gas composition. This flexible process design can produce layers with very different properties, e.g. hard/soft, hydrophilic/hydrophobic, to be combined to complex layer systems. Gradient layers are necessary to level out different properties between the substrate and layer e.g. a hard coating on a soft substrate like plastic. The gradient layers avoid damage to the layer from point loads, or a detachment due to stresses.

#### 2 Applications

All coating results presented in this study were obtained using laboratory systems at Fraunhofer ICT. The systems have a coating area of up to  $0.5 \text{ m}^2$  and 8 microwave sources with a maximum pulse power of 4 kW. The systems are equipped with evaporators and dosing systems for siloxanes and gases controlled by mass flow controllers. The temperature of the front and rear side of the substrate is measured with thermocouples and can thus be controlled. This is particularly important for coating temperature-sensitive materials, e.g. plastics.

The coating systems are equipped with powerful root pumps and dry-running screw pumps that allow high gas flows at working pressures of around 0.5 mbar [5]. The pre-pumping system is insensitive to dust generated in the process. The process parameters, e.g. gas composition, microwave power and working pressure, can be continuously changed, allowing layers to be deposited whose properties change gradually and can be adjusted over a wide range (Fig. 3).



Figure 3: PCVD-system at Fraunhofer ICT

#### 2.1 Transparent scratch protection on plastics

Plastic can be produced easily and inexpensively in almost any shape. It is light and often transparent. The major disadvantage is its soft and often sensitive surface, which can be protected with a glass-like layer (1). In order to achieve good scratch protection on a soft polymer substrate, a glass-like hard material layer with a thickness of at least  $3 \mu m$  is required.

On uncoated polycarbonate discs, 5  $\mu$ m thick scratch-resistant layers were deposited. At coating rates of more than 200 nm/s, the process takes less than 25 s. In order to achieve these rates, a reasonable amount of gas (HMDSO, oxygen) must be converted into the deposited glass layer. Because the working pressure is then relatively high, sufficient microwave energy is required to generate the large number of reactive species necessary for the high deposition rate.

The layers show high transparency (> 88 % transmission, approx. 0.5 % scattered light) and good

adhesion under a temperature change treatment in ice water and boiling water. The standardized Taber Abraser test (DIN 52347 or 53754) leads to slight changes (1 to 5 %) in haze (after 1000 rotations with friction wheel CS 10 F) - see Fig. 4.

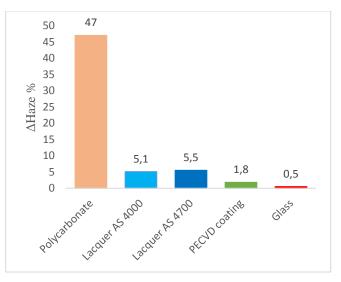


Figure 4: Taber-Abraser test results for different polycarbonate coatings

#### 2.2 Transparent corrosion protection of metals

The glass-like layer is also suitable for the corrosion protection of metals. In most cases, a layer thickness of  $1 \,\mu m$  is sufficient. To ensure that the layer adheres very well to the metal surface even at high temperatures of up to 500 °C, the surface must be subjected to a special plasma treatment immediately prior to coating. This pre-treatment is carried out in the PECVD system with the adapted gas composition. The gas composition is continuously adjusted to the composition required by the coating process. The pre-treatment is effective as the vacuum interrupted, is not and thus atmospheric contaminations are prevented.

Table 1 lists the main test results for aluminium, copper and steel. The corrosion protection of the coatings exceeds most standards for use in many applications.

This layer is suitable for damask knives (Fig. 5), protecting their blades against corrosion. The layer is transparent and reproduces the original structure of the damask steel.

Test	Test	Aluminium	Copper	Steel
	procedure			
Adhesion	Таре	~	$\triangleleft$	~//
	Cross Cut			$\vee$
Climate	Salt spray			
	test	672 h		500 h
	without	07211		500 11
	crack			
	Heat dry,		340 °C	500 °C
	16 h		340 C	300 C
	CASS with			~//
	crack, 48 h	$\otimes$		$\vee$
Hardness	Steel wool	$\triangleleft$	~//	~//
Abrasion	100 strokes		*	Ŵ

Table 1: Test results for metal coats

Corrosion protection also plays an important role in metal-plastic composite components. In particular, the combination of light, high-strength aluminium alloys with carbon-fiber-reinforced composites (CFRPs) immediately leads to contact corrosion and thus to component failure. Pure aluminium is coated with a thin inert oxide layer formed by atmospheric oxygen. In the case of highstrength aluminium alloys, such as those used in aircraft construction, this protective layer hardly forms [4]. The direct material connection between aluminium and CFRP is usually avoided with expensive connecting elements made of titanium, or weakening barrier elements made of glass or polymers. In this study, CFRPs were applied directly to plasma-coated and uncoated aluminium sheets. The prepared samples were salt spray tested. Four cycles were carried out. One cycle consisted of two hours of spraying 5 % NaCI with a pH value of 6.5 to 7.2 at a temperature of 35 °C. The samples were then sprayed for 1 hour with a degree of sharpness of 1. After this, the samples were stored for 166 hours at elevated temperature and humidity ( $T = 40 \ ^{\circ}C$  and 93 % humidity) for a total time of four weeks.

The results are shown in Fig. 6. It can be clearly seen that the glass-like layer protects the aluminium very well against corrosion. The aluminium/CFRP composite component adheres very well to the coated aluminium after 28 days (Fig. 6), while uncoated aluminium corrodes after a few days and the bond is dissolved. A direct connection between the metal and CFRP is possible with the PECVD corrosion protection coating.



Figure 5: Damask knives, blade coated with the PECVD corrosion protection layer (©Nesmuk)



Figure 6: Alu-CFK sample coated (left) und not coated (right) after SST

#### 2.3 Nanoporous adhesion layer

By varying the plasma parameters (working pressure, microwave power, gas flow, etc.), the glass layer can also be deposited porously. The porous structure forms independently of the substrate material and adheres very well to metal, glass, ceramic and many polymer surfaces. Figure 9 shows a scanning electron image of the porous layer. The porous layer grows in columns, and the distance between the columns is in the nanometer range. The individual columns are also porous.

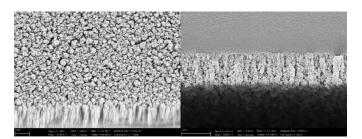


Figure 7: SEM image of the breaking edge of a substrate coated with a nano porous layer

The porous layer can be used as an adhesionpromoting layer between metals, glasses or ceramics and polymers. A liquid polymer, e.g. a molten thermoplastic or resin, is applied to the nanoporous surface, infiltrates the pores, solidifies and hooks there. Hooking the polymer into the porous structure significantly increases the adhesive strength of the compound. A glass sample was coated with a nanoporous adhesive layer and then infiltrated with liquid polypropylene (PP). A subsequent tensile test showed that the polypropylene (PP) breaks cohesively (Fig. 8).

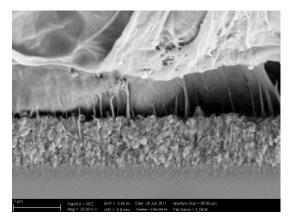


Figure 8: SEM image of the breaking edge of a nano porous layer infiltrated with PP after peeling

This nanoporous layer is also ideally suited for bonding plastic-metal components. A study confirmed the very good adhesive effect of the nanoporous layer compared to other surface pretreatment methods. For this purpose, aluminium, steel and brass plates with an area of 100 x 100 mm<sup>2</sup> and a thickness of 3 mm were produced. All the metal surfaces were blasted or coated with a nanoporous adhesive layer. Glass balls (70 µm in size) were used for the blasting, accelerated with an air pressure of 6 bar (~ 340 l/min). In addition, the aluminium plates were etched with phosphoric acid in accordance with DTD915B, and the steel surface was etched at 65 °C. The surface of the aluminium plates was then polished using a specially developed process. The coating formed on the steel surface was brushed off under clear water and the sample then dried in the oven at 120 °C for one hour. The brass surface was not etched because sodium dichromate which is usually used for etching brass, is toxic. Polyphenylsulfide platelets (PPS) were welded directly onto the samples prepared without any further adhesion promoter, and the tensile adhesion strength was then determined in accordance with DIN EN 24624 [7]. Figure 9 shows the results of the tensile adhesion tests for the various surfaces and pre-treatments.

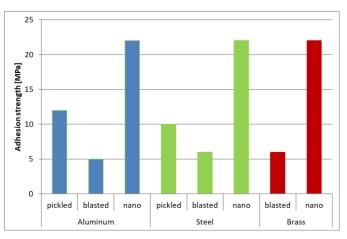


Figure 9: Results of the tensile test

As shown in Figure 9, adhesive strengths of about 22 MPa can be achieved with the nanoporous adhesive layer. This clearly exceeds the values of the other pretreatment processes. For example, 12 MPa were measured for etched aluminium and 10 MPa for brass. Blasting results in adhesive strengths of 5 to 6 MPa. It is particularly interesting that these high adhesive strengths are independent of the type of metal. This suggests that the adhesive strength is essentially based on the mechanical interlocking of the plastic in the pores, and less on chemical interactions at the interfaces. In contrast, the adhesive strength depends decisively on how well the plastic infiltrates the nanoporous layer. This is suggested by the results of a further study in which steel samples were coated with a nanoporous adhesive layer, placed in a injection moulding machine and overmolded with PPS. The achieved tensile strength of 47 MPa is significantly higher than in the previous welding tests, indicating that the injection moulding process leads to a better infiltration. Further investigations are necessary to clarify the complex interactions of the nanoporous layer with the infiltrated plastic and thus to optimize the tensile bond strength.

#### 3 Summary

Glass layers, applied with the microwave-generated PECVD process, refine surfaces and change their properties. By extending the pumping station, increasing the gas flow and microwave power, very high coating rates and thus short coating times of less than 1 min can be achieved for layer thicknesses of about 5  $\mu$ m, even on large surfaces. The quality of the coating is superior in comparison to lacquered protective coatings. Since the economic efficiency of PECVD coatings essentially depends on the coating time, these processes offer an economical and environmentally friendly alternative to conventional coating processes.

The glass layers protect metals very well against corrosion and are used industrially to protect damask knives. Even at very high temperatures of up to 640 °C, they exhibit good adhesion and excellent oxidation protection. They also provide very good electrical insulation and prevent contact corrosion in aluminium-CFRP composite components. This enables the simplified, direct and permanent joining of such composite components. Nanoporous glass layers are suitable for bonding materials such as glass, metal or plastics to polymers with high strength. In combination with the scratch and corrosion protection layers, these can be used to produce particularly adhesive, multifunctional coating systems.

#### For further readings

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#### About the Authors







Rudolf Emmerich has been involved in microwave technology for heating processes and plasma generation for more than 20 years. He heads the Mikrowellen and Plasma group at the Fraunhofer Institute for Chemical Technology. His work focuses on integrating microwaves polymer into processing and developing plasma processes

**Ralf Dreher** has been working with PECVD technology at the Fraunhofer Institute for Chemical Technology for more than 10 years. Currently he is project manager for the coating technology. His main focus is the development and optimization of glass-like protective and adhesive coatings.

**Marcel Laux** is a research associate at the Fraunhofer Institute for Chemical Technology. He is currently project manager for thermoplastic composites. His work focuses on hybrid metal-polymer materials

# Highlights of the 2018 Chinese National Conference on Microwave-Power Applications in Chemical Industries & Engineering

#### Junwu Tao

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IMPACIE 2018, the Chinese National Conference of Institute of Microwave Power Applications in Chemical Industries & Engineering, was held from 16th-18th August in Chengdu, China. This conference was organized by Sichuan University and the University of Electronic Science and Technology of China. It brought together 208 attendees including the top scientists and engineers to discuss the latest advances in the applications of microwave and high frequency technologies in chemical industries and This conference engineering. received 93 contributions and arranged 63 oral presentations. There were 35 universities, 7 academic institutions and 40 companies participating in the conference, with 33 companies in microwave techniques and 7 in chemical engineering. 12 Chinese companies joined the exhibition of this conference.

This conference got the support from other MAJIC confederation members: AMPERE, IMPI and JEMEA. Representatives from these associations were invited by Prof. Kama Huang, the conference chair, to give plenary talks. AMPERE was represented by Cristina Leonelli and Junwu Tao, IMPI by Roger Williams, and JEMEA by Nikawa Yoshio.

After the opening of ceremony, the morning session of the first day is was devoted to plenary talks given by both Chinese and international experts: "Microwave Desolvation Effect" by Prof. Kama Huang of Sichuan University,

- "Mechanical Research and Engineering Application of Microwave Chemical Reaction and Microwave Extraction" by Prof. Cheng Zheng of Guangzhou University,
- "An Overview of the European Group Research in the Field of High Power Microwaves" by Prof. Cristina Leonelli of University of Modena and Reggio Emilia,

- "The Microwave Power Research at INP Toulouse, France" by Prof. Junwu Tao of Toulouse University
- "Design, Development and Application of Microwave Fluidized Bed Drying Equipment" by Prof. Hongbing Ji of Sun Yat-sen University,
- "Competition, coexistence and development of solid-state sources and magnetrons" by Prof. Zhaotang Zhang of University of Electronic Science and Technology of China,
- "Medical and Biological Applications of Microwave and RF on the Basis of Tissue Characteristics" by Prof. Nikawa Yoshio of Kokushikan University,
- "Using Solid-state Sources to Bring New Levels of Control to Microwave Chemistry" by Dr. Roger Williams of American Ampleon Company,
- "Key Technology of Transparent and Collapsible Microwave Chemical Reaction Chamber" by Prof. Baoqing Zeng of University of Electronic Science and Technology of China,
- "New Progress in Microwave Metallurgy Industrialization Research" by Prof. Libo Zhang of Kunming University of Science and Technology.

During the afternoons of the first day and the second day the remaining oral communications, both from the Chinese researchers and industrialists have been organized in 4 separate sessions:

- Microwave Measurement and Simulation
- Microwave Power Devices and System
- Microwave and Matter Interaction Mechanisms of Microwave and Materials
- Microwave Chemical Applications

and the poster session has been organized in the exhibition area.

The gala dinner was organized on the 17th August and it was a memorable moment event. Shows representing Sichuan's traditional culture such as "Face Change" and "Long Beak Teapot" impressed all of the guests, while the Sichuan puppet show ended with the participation of Prof. Cristina Leonelli and Prof. Guoqing Dai, the representative of the Chinese Chemistry Association; the fight of their characters sparked the enthusiasm of everyone.

Several activities were organized for representatives of MAJIC confederation participating in IMPACIE 2018:

- a visit to the mountain Qingcheng, high place of Taoism, then Dujiangyan, ancient irrigation system originally built around 256 BC by the State of Qin as an irrigation and flood control project still in use today.
- Another visit to the Institute of Applied Electromagnetism (IAEM) at Sichuan University where Prof. Kama Huang presented his research projects around the microwaves power applications.
- A meeting to discuss some issues related to the organization of the 4th GCMEA- Global Congress on Microwave Energy Applications which is planned for 2020 in Chengdu.





Figure 1: Plenary session at 17th august morning



Figure 2: Plenary talk by Prof. Kama Huang of Sichuan University



Figure 3: Plenary talk by Prof. Cristina Leonelli of University of Modena and Reggio Emilia



Figure 4: Plenary talk by Prof. Zhaotang Zhang of University of Electronic Science and Technology of China

#### AMPERE Newsletter



Figure 5: Plenary talk by Prof. Yoshio Nikawa of Kokushikan University



Figure 6: Plenary talk by Dr. Roger Williams of American Ampleon Company



Figure 7: Photo with all attendees of IMPACIE 2018



Figure 8: Photo with local organization committee members of IMPACIE 2018



**Figure 9:** Visit of MAJIC members to Institute of Applied Electromagnetism (IAEM) at Sichuan University



Figure 10: Show of "Long Beak Teapot" during Gala Diner



Figure 11: show of "Face Change" during the Gala Diner



Figure 12: Nine-eye bridge night view taken after our working meeting on 4th GCMEA

#### About the author



Junwu Tao was born in Hubei, China, in 1962. He received his B.Sc. degree in electronics from the Radio Engineering Department, Huazhong (Central China) Universitry of Science and Technology, Wuhan, China, in 1982; the Ph.D degree (with honors) from the Institut National polytechnique of Toulouse, France, in 1988, and the Habilitation degree from the University of Savoie, France, in 1999. From 1983 to 1991, Dr. Tao

was with the electronics laboratory of ENSEEIHT, Toulouse, France, where he worked on the application of various numerical methods to 2- and 3-D problems in electromagnetics, and on the design of microwave and millimeter-wave devices. From 1991 to 2001 he was with the microwave laboratory (LAHC) at the University of Savoie, Chambéry, France, where he was an associate professor in electrical engineering and involved in the full-wave characterization of discontinuity in various planar waveguides, and in nonlinear transmission line design. Since September 2001 he is a full professor at the Institut National Polytechnique of Toulouse, where he is involved in the numerical methods for electromagnetics, microwave and RF components design, microwave and millimeter-wave measurements, and microwave power applications.

# IMPI's 52<sup>nd</sup> Annual Microwave Power Symposium

Molly. Poisant<sup>1</sup>, Sergey. Soldatov<sup>2</sup>

<sup>1</sup> Executive Director, International Microwave Power Institute (IMPI) <sup>2</sup> Karlsruhe Institute of Technology (KIT), Germany

The 52nd Annual Microwave Power Symposium (IMPI 52) was held in June 26-28, 2018 at the Hilton Hotel in Long Beach, California, USA (Fig. 1). It gathered 130 attendees from 16 countries. It became IMPI's llargest Symposium in more than a decade! A record number of 15 companies took part in the industrial exposition at IMPI 52: Ampleon, Dipolar, Ferrite Microwave Technologies, L3 Electron Devices, Macom, MKS, Muegge GmbH, NXP, PSC, SAIREM, SigmaPhi Electronics, Richardson Electronics, RFHIC, Rugged Monitoring and Wattsine. The symposium was sponsored by Muegge GmbH as a Gold Sponsor, Ampleon and Richardson as Silver Sponsors and Ferrite Microwave Technologies as a Bronze Sponsor. Shanghai Ocean University served as the Proceeding sponsor and Ampleon sponsored student travel scholarships to Candice Ellison, Louisiana State University; Tesfaye Bedane, University of Salerno, Yoon-Ki Hong, Washington State University and Mine Ozcelik, University of Munich.



Figure 1: Gallery Ballroom Hilton hotel.

The symposium program listed 65 oral and poster presentations on a range of topics including: Solid State, Food & Agriculture, Novel Microwave Applications, Microwave Packaging, Modeling and CAD, Food Processing, Microwave Chemistry, Industrial Processing, Microwave Equipment, Food Safety and Microwave Ovens and Material Handling Up.

The symposium began with the Short Course offered by Dr. Vadim Yakovlev: "Introduction to Multiphysics Modeling in Microwave Power Engineering" and a spotlight session including 5 papers and a panel discussion, chaired by IMPI President, Bob Schiffmann. Attendees ended the first day with a Welcome Reception in the Ballroom of Hilton Hotel.

Wednesday started with keynote presentation on "Microwave Food Processing - Reinventing Packaged Food" given by Lora Spizzirri, 915 Labs LLC, USA and continued by Exhibitor Spotlight session. Thereafter the Symposium continued in parallel sessions on Microwavable Packaging, Modeling and CAD, Microwave Equipment and Novel Microwave Applications. In the afternoon, the Symposium featured parallel sessions on Material Handling-Up and Food Safety. Here, we like to distinguish the talk of M. Garuti "Microwave Assisted Manufacturing of Package-less Coffeetablet: Magnetron Vs Solid State" where the importance of source frequency control in resonant applicators was underlined. Two invited talks of K.-M. Baumgaertner "CiMPAS - A Novel Approach for Fast Inline Microwave Pasteurization and Sterilization" and K. Werner "Solid-state RF Energy - Advancements in Industrial Applications and Market Opportunities" end the presentations given on the second day. Thereafter the IMPI Business Meeting took place where two Fellow Awards were granted to Dr. Vadim Yakovlev of Worcester Polytechnic Institute and Dr. Raymond Boxman from Tel Aviv University (see Fig. 2). These are the first fellow awards given since 2013. In the evening the attendees enjoyed a group dinner at a local restaurant L'Opera with Italian cuisine.



**Figure 2:** Fellow Awards were granted to Dr. Raymond Boxman (left) and Dr. Vadim Yakovlev (right)

Beginning Thursday morning Brad Hoff, AFRL, USA gave a keynote address "Millimeter Wave Interactions with High Temperature Materials and their Application to Power Beaming". After that the audience was divided between two sessions: Microwave Assisted Chemistry and Food & Agriculture. Here we like to mark out the talk of Graham Brodie where the theory of evanescent mode finds a nice realization in the evanescent field applicator for soil heating and already demonstrated its higher efficiency as compared to direct irradiation onto soil surface. Another interesting presentation was given by Huixin He, Rutgers University, USA, where the advantage of microwave treatment by manufacturing of graphene structures is clearly presented. The formation of nano-holes and sufficient control of zigzag edges of graphene structures during microwave assisted oxidation are highly preferred for their use in catalytic applications.

15 students competed in the Student Competition. Best Oral Presentation went to Hermine Tertrais (Ecole Centrale de Nantes, France); Best Poster Presentation went to Chuting Gong (Shanghai Jiao Tong University, China). Honorable Mention in the Oral Presentation category went to Candice Ellison (Louisiana State University, USA) and Humayun Kabir (University of Melbourne, Australia).

Students winners and honorable mentions won a cash prize, a certificate and a one year student membership to IMPI.

During the closing ceremony on June 28<sup>th</sup>, the location for IMPI 53 was announced. The 53rd Annual Microwave Power Symposium (IMPI 53) will take place from June 18 - 20, 2019 at Ceasars Palace in Las Vegas Nevada, USA. Dr. Graham Brodie of the University of Melbourne and Mr. Roger Williams of Ampleon will serve as the Cochairmen on the Technical Program Committee. Dr. Ulrich Erle of Nestle will serve as the Chairman of the Food Science & Technology Program Committee.

#### About the authors



**Molly Poisant** has served as the Executive Director of the Int'l Microwave Power Institute (IMPI) since 2010. She has over 20 years of experience in event operations, business development, legislative affairs and sponsorship sales having worked for two former Governors and

several U.S. and international technology conferences. She received her Bachelor's degree in Political Science from Longwood University.



**Sergey Soldatov** received the Dipl.-Ing. degree in experimental nuclear physics and plasma physics from MEPhI (Moscow) and Ph.D. degree from National Research Center "Kurchatov Institute". From 1994 to 2005 as an expert in microwave diagnostics he was involved in fusion plasma experiment at TOKAMAK-10 (Moscow) and later (2005-2011) at

TEXTOR (Jülich, Germany). Since 2011, Sergey Soldatov is a leading researcher in the Institute for Pulsed Power and Microwave Technology at Karlsruhe Institute of Technology (KIT), Germany. He specializes in design and development of microwave applicators for thermal material treatment as well as microwave sustained plasma systems. His research interest covers also plasma diagnostics, antenna systems, dielectric characterization and multi-physics simulations.

## Ricky's Afterthought:

# What is Gravity?

A.C. (Ricky) Metaxas

Life Fellow St John's College Cambridge UK Email: <u>acm33@cam.ac.uk</u>

Well before indulging in questions that we cannot answer let us consider the obvious manifestations of gravity. All objects exert gravity on other objects. The larger the objects are and the shorter the distance between them the stronger the pull on each other.

The four forces that are inherent in the Standard Model of the Universe are the strong and weak nuclear forces, the electromagnetic force and gravity. It is well known that Einstein in his relativity papers postulated that space is inherent to gravity as it curves around large objects. Although gravity is weak compared to the other three forces, unlike the other three it is only positive and therefore cumulative and cannot be cancelled out by positive or negative parts as in the other three.

Newtonian gravity, such as in the solar system has been measured and is well understood, however, we know that the expansion of the universe is accelerating so somehow we must study gravitational forces over large distances. Is gravity weaker at interstellar distances or is the accelerated expansion driven by an unknown energy, termed dark energy? One idea is that quasars may hold the answer and scientists are looking at multiple images of the brightness of these quasars to see if it's constant, and subsequently measure the length of time the light takes to get to Earth. One estimate is that one needs to observe over many quasars to get sufficient information but only a handful of these are found to date.

Another major project currently under way is the Illustris cosmological simulation project, which is an ongoing series of astrophysical simulations run by an international collaboration of scientists. The aim is to study the processes of galaxy formation including dark energy and dark matter and combine



it with a comprehensive physical model of the Universe.

How about the notion that black holes and dark matter are linked? Black holes are massive and as such exhibit a strong gravitational force.

Another study to unravel the mysteries of gravity is to consider the Cosmic Microwave Background (CMB) radiation which is the residual electromagnetic background of the Big Bang which engulfs the entire Universe (see Afterword article AMPERE Newsletter March 2012 Issue 72). In 1964 two engineers, Arnold Penzias and Robert Wilson at New Jersey USA, set out using their Holmdel Horn Antenna to observe the galaxy through the invisible light that emanated from it. However from the very start they realised that they were picking up some background interference which masked what they were trying to measure. Having checked the entire equipment over and over again they could not get rid what they thought was simply a troublesome "noise" signal. The only explanation left was the very unlikely event of this radiation coming from outside our galaxy. Eventually physicists realised that this was the remnant of radiation originated immediately after the Big Bang some 13.75 billion years ago. Today data from Planck's CMB are used to understand the amount of dark matter there is in the Universe and specifically whether the amount changes over time. Planck is a European Space Agency space-based observatory observing the Universe at wavelengths between 0.3 mm and 11.1 mm (corresponding to frequencies between 27 GHz and 1 THz), broadly covering the far-infrared, microwave, and high frequency radio domains.

Finally, the remarkable fact is that after hundred years since Einstein developed his theory of

relativity stating, that gravity arises from the curvature of space and time, the data fit that theory. Cosmologists are holding their breath at the thought that in the future data may reveal the need to modify Einstein's theory. However, bear in mind that even if that happens does not prove that Einstein was wrongafter all the moon landings were based on Newtonian mechanics not relativity. We simply have to adapt our existing theories to fit the new findings.

Does this explain what gravity is? The emphatic answer is No!

The above was extracted from the following article: https://www.alumni.cam.ac.uk/news/a-force-to-bereckonedwith?utm\_medium=email&utm\_source=EN1117

Upcoming Events

**Summer School in High Energy Processing Ultrasound & Microwave Technologies** – 26<sup>th</sup> -28<sup>th</sup> June 2019, Cracow, Poland.

Sponsored by AMPERE and the Euopean Society of Sonochemistry (ESS), the Faculty of Chemical Engineering and Technology, Cracow University of Technology in Poland host this interdisciplinary summer school which is limited to 40 participant. The program and registration details are available on the following Website: <u>http://school2019.chemia.pk.edu.pl/</u>

#### 17th International Conference on Microwave and High Frequency Heating: AMPERE 2019

9<sup>th</sup>-12<sup>th</sup> September 2019, Valencia Spain

The 17th International Conference on Microwave and High Frequency Heating: AMPERE 2019 is the largest event in Europe dedicated to scientific and industrial applications of microwave and radiofrequency power systems. The conference presents the status and trends in the multidisciplinary fields of microwave and radiofrequency heating, dielectric properties, material processing, high power systems and technologies. The AMPERE conference is a unique opportunity for the presentation and discussion of the most recent advances in the microwave technology and its applications. The conference provides many opportunities to researchers and engineers from academia and industry to exchange innovative ideas, networking, discuss collaborations and to meet with international experts in a wide variety of specialities of microwave and high frequency technologies at both scientific and industrial scale.

Website: http://ampere2019.com



### About AMPERE Newsletter

AMPERE Newsletter is published by AMPERE, a European non-profit association devoted to the promotion of microwave and RF heating techniques for research and industrial applications (<u>http://www.AmpereEurope.org</u>).

# Call for Papers

AMPERE Newsletter welcomes submissions of articles, briefs and news on topics of interest for the RF-andmicrowave heating community worldwide, including:

- Research briefs and discovery reports.
- Review articles on R&D trends and thematic issues.
- Technology-transfer and commercialization.
- Safety, RFI, and regulatory aspects.
- Technological and market forecasts.
- Comments, views, and visions.
- Interviews with leading innovators and experts.
- New projects, openings and hiring opportunities.
- Tutorials and technical notes.
- Social, cultural and historical aspects.
- Economical and practical considerations.
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AMPERE Newsletter is an ISSN registered periodical publication hence its articles are citable as references. However, the Newsletter's publication criteria may differ from that of common scientific Journals by its acceptance (and even encouragement) of news in more premature stages of on-going efforts.

We believe that this seemingly less-rigorous editorial approach is essential in order to accelerate the circulation of ideas, discoveries, and contemporary studies among the AMPERE community worldwide. It may hopefully enrich our common knowledge and hence exciting new ideas, findings and developments.

Please send your submission (or any question, comment or suggestion in this regard) to the Editor in the e-mail address below.

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