Graphene standardization: The lesson from the East

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How do scientific ideas become market products? There is probably no single pathway for such transformation. And yet, there are certain similarities in the way how advanced materials evolve from laboratory studies to being used in technology. Common steps in such progress are the enhancement of useful properties, development of the production methods, creation of industrially-relevant modification of the material itself and its fabrication process. The reason in the emergent similarities in the pathway to market is the established relation between materials supplier and the final product manufacturers. A dramatic role in such relations is played by industrial standards. The later can help, but also, if incorrectly developed, can stumble the final product development. We will study the process of commercialisation of graphene, its transformation to commodity and the emerging graphene standardisation efforts.

Keywords: Graphene; 2D materials; Standardization; Nanomaterials; Applications; Commercialization

Transition of any new idea, technology, material or device from the research laboratory to industry and mass production is always an exciting, but lengthy and difficult process. The major reason for this is the very difference in approaches: research values plurality of methods and models, whereas industrialisation requires unified methodologies. Very often such unified approaches start with the formation of industrial standards.

The later can help, but also, if incorrectly developed, can stumble the final product development. We will study the process of commercialisation of graphene, its transformation to commodity and particularly highlight the basic ‘elements’ to be considered in the emerging graphene standardisation efforts.

Concerns in commercialization

These days graphene [1] firmly established itself as one of the most exciting and widely-used material for research and technology [2]. One of the reasons of such popularity is the versatile nature of graphene. What we call graphene is in fact a generic name for a broad range of materials with different structure, morphology, sometimes even chemical composition. Extremely confusing for a non-expert, the properties of different “graphenes” are strongly dependent on the preparation method. But it is exactly this variety of properties which allows graphene to be used in

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many applications, covering a range of technologies, costs, ways of manufacturing.

Depending on the application and technology, different forms of graphene and different production methods are used. Generally, graphene production can be divided into two large classes: bottom-up and top-down [2,3]. Bottom-up methods include epitaxial growth of graphene, such as growth on copper [4] or silicon carbide [5] among others. These methods allow formation of continuous polycrystalline graphene films (though wafer-size graphene monocrystals have also been demonstrated [6] and are generally used for electronic, photonic and transparent conductive coating applications [7]). Top-down methods (used to produce graphene for energy, printable electronics, composite and other applications), instead, base on exfoliation (mechanical, chemical, electrochemical and other) of graphite into various flake products: graphite nanoplatelets, graphene platelets, graphene quantum dots and so on [8]. Furthermore, chemical and electrochemical exfoliation can result in chemical modification of graphene, increasing the range of graphene-based materials even further [9]. We refer to recent reviews for a full description of graphene-based materials [10–12].

The wide range of production methods causes a great confusion for technologists and product engineers. Such a variety of forms is responsible for the large number of inconsistent and even contradictory results in graphene applications. To achieve equally firm status in technologies and manufacturing as it has in scientific world, graphene should be able to build-in itself into the existing technological and supply chains, with all their rules and regulations. Manufacturers of graphene materials and products need to have a mechanism of efficient communication with the consumers (as well as between themselves) in order to establish a system which allows fast and efficient modification of the properties, which will not result in significant changes of the established technological chains.

To this end, the whole industry would benefit enormously from standards. These standards, however, need to be application facing, application friendly and application specific in order to help, rather than stall the progression of technology.

Examples of successful graphene applications

Despite the difficulty in translation of graphene from lab to industry, there are a number of successful cases of implementation of graphene in advanced products on a large scale. Usually it happens where both the manufacturer of the final product and the graphene supplier are both willing to invest time and money into the development of bespoke graphene material specifically for the particular application. Typical problems which have to be resolved during such a process are ensuring reproducibility of the parameters of the new material, as well as its compatibility with the production processes and technological restrictions already established at the manufacturer side. Such process of interaction between material supplier and the final product manufacturer usually results not only in very strict definition of the parameters of the supplied materials (which often translates into industrial standards) but also into requirements for the production process. Here we give two examples of such applications.

The first one is related to the high thermal conductivity of graphene. The reported value [13] of 5,000 W/m·K is probably the highest among any materials. At the same time, thermal spreading and dissipation is among the most important unsolved problems in modern electronics. Miniaturization of transistors (which allows higher transistor density) and increase in the clock frequency lead to progressively larger thermal dissipation in modern electronics, which leads to a strong demand for efficient cooling. Materials with high thermal conductivity are actively used in electronics for efficient heat distribution. Thin graphitic films, obtained by graphitization of polyimide, have been successfully used for these purposes for a number of years by many companies. Such graphitic films, however, have two major drawbacks: they are not very flexible and their thermal conductivity reduces dramatically with increasing thickness (from ~2,000 W/m·K in 10 μm thick films to 700 W/m·K in 100 μm thick samples) [14].

Graphene films obtained by reduction and compression of graphene oxide paper [15], solve these problems and it has already been proposed for the use in chip cooling [11]. Such graphene paper remains flexible and its thermal conductivity practically doesn't depend on the thickness. This leads to the increasing use of such paper for electronic applications. Thus, Huawei Inc. believes that the graphene film can provide an excellent solution for higher heat dissipation needs in the 5G era [16].

Clearly, the material manufacturers had to do a lot of work in terms of matching their products to the requirements of the final product manufacturers. Apart of the obvious – thermal conductivity (which has to be measured according to the industrial standards) – there are many other parameters which need to be controlled and regulated: electrical conductivity, thickness, thermal expansion, working temperatures, etc. And there are a number of other parameters, not so obvious ones. For instance, the films need to be cut into shapes, which brings requirements in terms of shedding of flakes, which, in turn, sets requirements for the size of the flakes used in the production of such films.

The second example concerns the use of graphene as electrode in lithium (Li) ion batteries. The use of graphene in energy storage was on the top of the agenda from the very beginning of graphene saga. Indeed, many studies have proved that, the large specific surface area, good conductivity and chemical stability of graphene bring advantages in certain aspects, when graphene is used as main or auxiliary materials in batteries. For instance, it was predicted that graphene can be used as the anode of Li ion batteries to provide capacity twice larger than graphite [17]. Nevertheless, compared with the stable voltage platform corresponding to the intercalation of Li ions into graphite, the adsorption and side reactions of Li ions on graphene is a very complex process, involving defects and chemical doping [18]. In fact, the low Coulomb efficiency and questionable stability (caused by the formation and evolution of solid electrolyte interface on graphene) makes it almost impossible for this material to be used as the main component in the existing Li ion batteries [19]. Thus, the main usage of graphene in energy applications is as a conductive additive for electrodes.

Essentially, the two-dimensional (2D) nature of graphene platelets as well as their flexibility have contributed to its advantages in terms of improving the electric conductivity of electrode materials by wrapping-up the active materials. Especially the superior performance is obvious when compared to currently used 1D carbon nanotubes and, in particular, carbon black, which mostly rely on the point-to-point contact [20] in terms of electrical conductivity. But some studies indicated the possible blocking of Li ions by the 2D platelets, thus certain kind of matching between the size of graphene and that of electrode particles or pore engineering in graphene seems important [21], which immediately gets reflected in the required size distribution of graphene flakes. Another requirement is graphene dispersibility in specific solvents already used in the battery industry. Once the dispersibility and control of the size distribution issues are overcome, one can expect booming of graphene-based conductive additive in the market not limited to Li ion batteries since the use of graphene may greatly decrease the required amount of other carbon-based additives. Already now BYD Battery Inc. purchases hundreds of tons of suspensions containing graphene platelets every year, to improve the performance of $LiFeO_4$ cathode [22]. This industry is still in infancy, with many battery companies still experimenting with the composition of their conductive additives. In order to streamline this process even greater control is required on the characterization of graphene conductive additives in terms of flake size and thickness distribution as well as their chemical composition and impurities. Furthermore, other parameters, such as the viscosity of premixed graphene slurry as well as their stability should be controlled by material producers.

The similar situation applies to the applications of graphene materials for another energy storage device – supercapacitors. The huge specific surface area of graphene makes one to expect very high adsorption of ions on this material, leading to an energy storage capacity close to the lead acid battery but with much shorter charging/discharging time. After years of intensive studies, however, researchers have solved the big challenge to simultaneously maintain the huge surface area and a reasonable density, and the latter is critical to convey the superior materials merits to outstanding device performance as the spare space in low density electrode would have to be filled by liquid electrolyte, intrinsically lowering the energy density per device [23]. Thus, densified graphene electrodes while keeping the high ion storage and transport ability would be valuable to the future energy storage market, in addition to the cost of graphene. Capillary drying of graphene hydrogel presents a possible solution for densified electrodes and produces compact supercapacitors, although more detailed consideration of ionic behavior shall be taken in such porous materials [24]. The same technology has now extended to Li ion batteries highlighting the challenge of storing more energy in more limited space [25].

Graphene standards

Even the above two examples show the complex landscape of graphene applications, which brings a very difficult question of standardization of this material, as depicted in Fig. 1. Clearly the large corporations mentioned cannot function without excellent reproducibility of the parameters of the materials used, but the parameters of interest would be very different in the two cases.

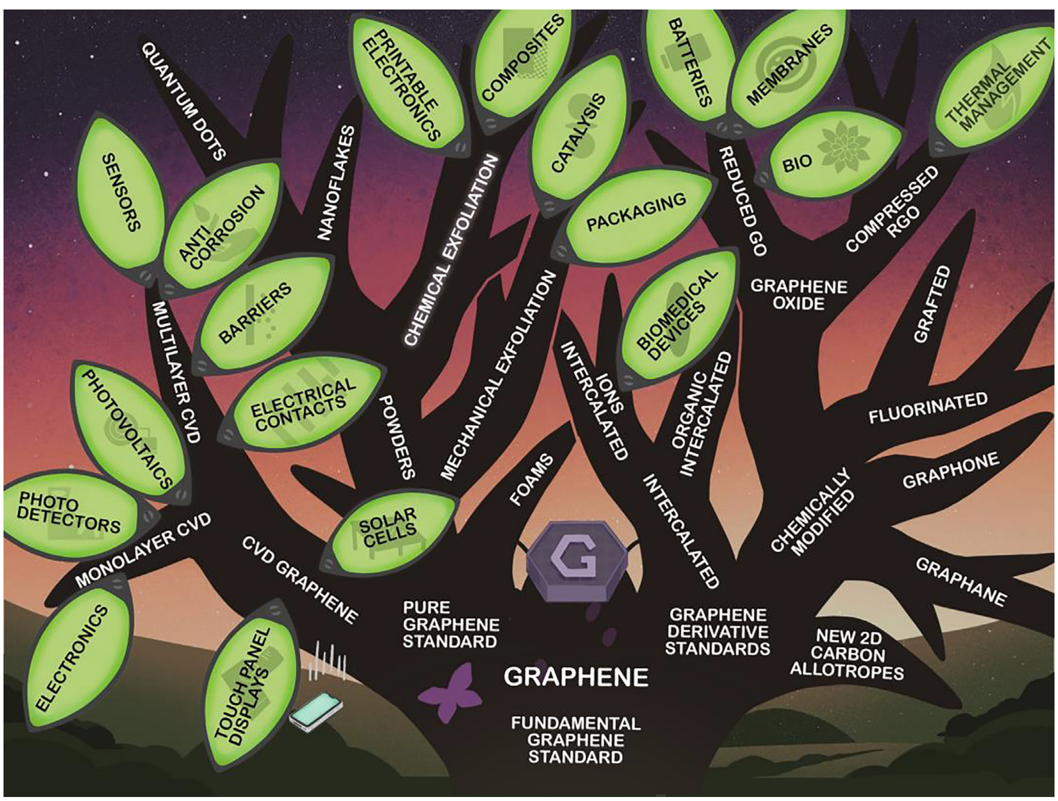

FIGURE 1

Hierarchy of graphene technologies and standards.

The high-level standard, which simply defines what is graphene, would do little to help industrial companies to narrow down the particular properties required to fulfill certain functions in a specific device. One well-studied situation is the chemical functionalization of graphene, e.g., with hydrogen, oxygen, fluorine, etc., leading to materials called hydrogenated graphene (or graphane), graphene oxide, fluorographene and so on. The decoration or doping of adatoms on graphene would benefit certain applications but has made the definition of graphene more complex. A wide range of other graphene derivatives have broadened the applications of graphene family but surely would confuse people what graphene is in terms of standardization.

Furthermore, industrially-friendly standards need to provide specific protocols for the controls of standardized parameters. Defining graphene as exactly one layer thick fabric would do little help to industrial players as they would not have access to the measuring tools which can access thickness of individual graphene crystals at fast enough pace. At the same time, one can form a different frame of parameters which would define graphene equally well in industrially-friendly way. For instance, for the sake of fast developing field of Li batteries, one can define specific surface area, rather than thickness of individual crystallites; amplitude, shift and broadening of the Raman peaks, rather than the concentration of impurities and defects and so on.

There are more considerations to this. Introduction of any new materials into real products are associated with multidimensional Pandora box of problems: we need to think about licensing of new materials, toxicological and environmental aspects, and so on. All of those issues are closely related to the standards introduced, and such introduction need to be done extremely responsibly. Even higher level issues, such as marketing (which products can be called graphene products and which cannot) are dependent on standardization.

Thus, we envisage that there should be a well-structured hierarchy of patents, from the very top where general definitions of what is graphene and what are graphene-like materials to the very bottom where applications-related standards (together with the methodology and protocols of their evaluation) would help to address the specific properties of graphene. We would like to stress that this structure of standards need to be coherent and hierarchical, so no contradiction is introduced at this stage. Creation of such a hierarchy (together with a set of industry-friendly protocols which would help enforcement of such standards) is a very complex, though extremely important, task.

In recent years, international standards organizations such as ISO (ISO/TC 229 Nanotechnologies) and IEC (IEC/TC 113 Nanotechnology for electrotechnical products and systems) have already issued several graphene-related standards [26,27]. Countries with active graphene research, such as UK, USA, Japan, Korea and China, are actively participating in the projects of developing international standards, and dozens of new standards are being schemed. Organizations, academia and government in European Union have made crucial efforts to standardization of graphene, in which graphene flagship standardization committee (GFSC) plays an important role, to establish normalization document of graphene materials towards commercialization. Over 35 projects of standards and technical specifications has been proposed or is being developed in GFSC, including terminology, measurement and characterization methods, normalization to graphene suppliers and buyers, and so on.

These, however, are very high-level instructions, which are not always industry-friendly. In the vacuum created, industrially developed countries are quick into development of lower level standards relevant for specific technologies. Driven by the powerful industrialization of graphene, China has published a number of graphene-related standards not only at the level of central and local governments, but also in relevant industries and market directions [28], to provide guidance and reference for graphene to become a product and enter the market (see Table 1).

As can be seen from the table, formally published and publicly available graphene standards cover a wide range of aspects, from terminology to test characterization methods and products. However, the standardization process is far from smooth, and the debate is partly due to the different views held by academia and business circles, reflecting the complexity of creation of a coherent hierarchical structure of standards.

For instance, in 2017, ISO and IEC jointly released a standard for graphene-related basic terms, *Nanotechnology Terminology Part 13: Graphene and Related Two-Dimensional Materials*, which was also adopted by the China Technical Committee for Standardization of Nanotechnology (SAC/TC 279) [29]. This standard draws a clear line under the discussion of what is graphene, defining it as exactly a single layer of carbon atoms. As a consequence, bilayer/trilayer/few-layer graphene should be called "two-dimensional materials containing several graphene layers", but not graphene. This simple move alone produced quite a steer on the market, as many of graphene-related products would contain material with more than one layer of graphene [30].

The aforementioned standard represents the opinion that a carbon-based material shouldn't be called graphene only because it is useful, including many graphene derivatives mentioned above. In contrast, another standard on *Graphene Materials Terminology and designation* appears to be quite loose, which is formulated by representatives of academic and business circles organized by several graphene alliances in China [31]. The standard has come up with a general term 'graphene material' for materials and products consisting of no more than 10 layers of graphene, either individually or tightly packed. Thus, 'graphene-based material' here includes not only 'monolayer graphene', 'bilayer graphene', but also 'graphene oxide', 'hydrogenated graphene' and so on. This standard focuses more on the functionality of the final product, asking a question if the product with added graphene-based materials gains new properties or not? Depending on the answer to this question the final product may or may not use the title of graphene in the name.

Obviously, the categories and market sizes of graphene products defined by the above two standards would vary greatly, which would partly explain the huge difference in the estimation of graphene market space in the future (*e.g.*, by 2025) given by different market studies [32].

Understanding that introduction of every new standard would create initial havoc in the established system and might break the *status quo*, we still think that the enforcement of standards is extremely useful and necessary in the long term, considering it is done methodically and coherently.

TABLE 1

Publicly available standards related to graphene.

Source	Tile of Standard	Publication Organization	Standard Number	Classification	Statue
International	Nanotechnologies—Vocabulary—Part 13: Graphene and related two-dimensional (2D) materials	International Organization for Standardization (ISO)	ISO/TS 80004-13:2017	Terminology	Accept payment https://www.iso.org/standard/64741.html
	Nanotechnologies—Matrix of properties and measurement techniques for graphene and related two-dimensional (2D) materials		ISO/TR 19733:2019	Measurement/ Method	Accept payment https://www.iso.org/standard/66188.html
	Nanomanufacturing – Key control characteristics – Part 6-4: Graphene - Surface conductance measurement using resonant cavity	International Electrotechnical Commission (IEC)	IEC TS 62607-6-4:2016	Measurement/ Method	Accept payment https://webstore.iec.ch/publication/25950
China/national	Nanotechnologies—Vocabulary—Part 13:Graphene and related two-dimensional (2D) materials	National Technical Committee 279 on Nanotechnology of Stardardization Administration of China	GB/T 30544.13-2018	Terminology	Accept payment https://www.chinesestandard.net/China/Chinese.aspx/GBT30544.13-2018
	Nanotechnologies – Determination of specific surface area of graphene materials—Methylene blue adsorption method		GB/Z 38062-2019	Measurement/ Method	Open http://www.cssn.net.cn/cssn/front/110526250.html
	Nanotechnologies – Quantitative analysis of the surface oxygen functional groups on graphene materials—Chemical titration method		GB/T 38114-2019	Measurement/ Method	Open http://www.gb688.cn/bzgk/gb/newGbInfo?hcno=05223E5FA0DF26920BA548B964F0928E
	Graphene Zinc Coatings	Ministry of Industry and Information Technology, China	HG/T 5573-2019	Application	Open http://std.samr.gov.cn/hb/search/stdHBDetailed?id=9F25957A194447DAE05397BE0A0A0983
China/ associations	Graphene-enhanced extreme pressure lithium grease for construction machinery	Zhongguancun Standardization Association, Beijing, China	T/ZSA 74-2019	Application	Accept payment http://www.ttbz.org.cn/StandardManage/BuyDetail/32676/
	Graphene modified rigid electric heating pad		T/ZSA 73-2019	Application	Accept payment http://www.ttbz.org.cn/StandardManage/BuyDetail/32675/
	Graphene modified flexible electric heating film		T/ZSA 9001.01-2017	Application	Private http://www.ttbz.org.cn/StandardManage/Detail/21754/
	Epoxy graphene zinc primer	China Coating Industry Association	T/CNCIA 01003-2017	Application	Open http://www.ttbz.org.cn/StandardManage/Detail/22146/
	Waterborne graphene electromagnetic shielding coating for architecture		T/CNCIA 01004-2017	Application	Open http://www.ttbz.org.cn/StandardManage/Detail/22147/
	Graphene heating tiles	Guangdong Enterprise Innovation and Development Association, Guangdong Province, China	T/GDID 1012-2019	Application	Private http://www.ttbz.org.cn/StandardManage/Detail/32376/
	Graphene hollow yarn fabric with antibacterial and deodorant	Nantong textile industry association, Jiangsu Province, China	T/NTTIC 022-2019	Application	Private http://www.ttbz.org.cn/StandardManage/Detail/32223/
	Graphene materials terminology and designation	Zhongguancun Huaqing Innovation Alliance of the Graphene Industry, Beijing, China	T/CGIA 001-2018	Terminology	Accept payment http://www.ttbz.org.cn/StandardManage/BuyDetail/23102/
	Determination of silicon content in graphene materials—		T/CGIA 013-2019	Measurement/ Method	Accept payment http://www.ttbz.org.cn/StandardManage/BuyDetail/30289/

(continued on next page)

TABLE 1 (*CONTINUED*)

Source	Tile of Standard	Publication Organization	Standard Number	Classification	Statue
	Molybdenum blue spectrophotometry				
	Determination of metallic elements in graphene materials—Inductively coupled plasma emission spectrometry		T/CGIA 012-2019	Measurement/ Method	Accept payment http://www.ttbz.org.cn/StandardManage/BuyDetail/30288/
	Test method of iodine adsorption number for graphene materials		T/CGIA 011-2019	Measurement/ Method	Accept payment http://www.ttbz.org.cn/StandardManage/Detail/29810/
	Guidance on naming of products containing graphene materials		T/CGIA 002-2018	Measurement/ Method	Accept payment http://www.ttbz.org.cn/StandardManage/BuyDetail/23101/
	Graphene-enhanced extreme pressure lithium grease for construction machinery		T/CGIA 31-2019	Application	Accept payment http://www.ttbz.org.cn/StandardManage/BuyDetail/30287/
	Graphene materials conductive suspension for use in lithium ion battery application		T/CGIA 032-2019	Application	Private standard@c-gia.org
	Electric infrared radiant heating film made by printing ink based graphene materials		T/CGIA 030-2017	Application	Private http://www.ttbz.org.cn/StandardManage/Detail/4045/
	Test method for identification of graphene materials in fibres–Transmission electron microscope (TEM) method	China National Textile And Apparel Council	T/CNTAC 21-2018	Measurement/ Method	Private http://www.ttbz.org.cn/StandardManage/Detail/25301/

*These data are updated to Feb. 2020.

One example of such downstream standard which inherits the more general standards, is the industry standard of *Graphene Zinc Coatings* (HG/T 5573-2019), published [33] by the Ministry of Industry and Information of China and which has been implemented on July 1, 2020. The standard comprehensively describes the requirements for graphene-based product, quality control, packaging and storage when graphene and zinc powder are used together to prevent corrosion of steels. The standard was proposed by the China Petroleum and Chemical Industry Federation and drafted by a group of graphene manufacturing enterprises, coatings manufacturing and application enterprises, and related research institutes. Certainly, it is not easy to give comprehensive and detailed information of graphene in the standard (as an example, please refer to the appendix for the standard of *Graphene Zinc Coatings*).

How long it might take to create a coherent system of standards? It is very difficult to predict, but a lesson from the already mentioned use of carbon nanotubes as conductive additives tells us that the process is not fast at all. After almost 30 years of research and development, the market of carbon nanotube conductive additives has occupied about 20% of the total additive powders for Li ion batteries in China. More than 1400 tons of carbon nanotubes are used in batteries every year. But the first international standard related to carbon nanotube suspensions (ISO/TS 19808:2020, Nanotechnologies – Carbon nanotube suspensions – Specification of characteristics and measurement methods) was published by ISO only in March 2020.

With the experience from carbon nanotubes, the standardization of graphene-based conductive additives has been proceeding more quickly. Although the estimated market share of graphene based conductive additives in China at the moment is only ~15% of that of carbon nanotubes, a group standard (T/CGIA 032-2019) named 'graphene materials conductive suspension for use in Li ion battery application' has been implemented in Dec 2019, drafted by a long list of Li ion batteries-related companies and researchers from institutes of Chinese Academy of Sciences. Instead of regulating the graphene-containing composite suspensions, *e.g.*, with carbon black or carbon nanotubes or both, as commonly used in the current market, the standard tries to encourage a system of features of 'pure' graphene suspensions, such as the layer number and particle size of graphene platelets in the suspensions, other chemical components allowed in the suspensions, and the electrical resistivity of the cathode electrode membranes made from the additive. The standard highlights that, the final users may mix such 'standardized' graphene suspensions with other necessary components according to their own needs. The key 'elements' to be considered in graphene-based conductive additives for batteries are drafted in Fig. 2.

One of the complications associated with graphene is that it is still a developing technology with production and characterization methods constantly adapting to the recent scientific advances and requirements from industry. Beyond graphene, recent developments in the field of van der Waals heterostructures [34–35] open even broader opportunities application-wise [36], but thus creating additional challenges in standardization.

As a conclusion we would like to suggest creation of a coherent, hierarchical structure of standards with industrial standards being accomplished by comprehensive methodology of controlling materials properties as well as of the parameters of the final products. This still calls for stronger communication between academia and industries in order to achieve more scientific understanding of the roles of graphene in particular products, which would allow one to avoid using the name of graphene in gimmick products. The role of government and

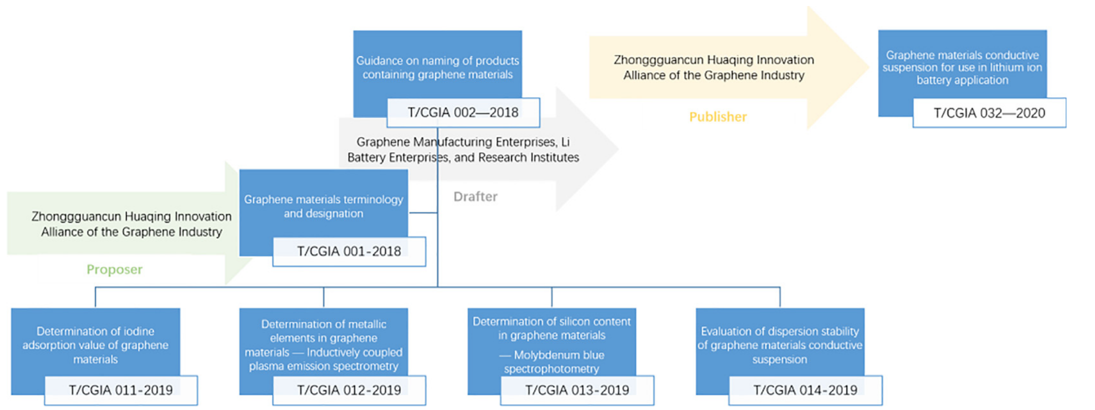

FIGURE 2

A hierarchy consisting of several group standards by and driving bodies towards the graphene suspension standards for Li ion battery application.

standardization organizations in this process should be strengthened, though every new policy introduced should take into account the interest of industry, where the real innovations take place. For instance, a 'new material insurance' in China is expected to promote the commercialization of graphene materials [37,38]. We believe that, when a benign ecology is formed based on the interactions between scientific community, business community, government and the general public, graphene will contribute to the development of human civilization as many new materials which have been introduced in history.

CRediT authorship contribution statement

Yanwu Zhu: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing - original draft, Writing - review & editing. **Bill Qu:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing - original draft, Writing - review & editing. **Daria V. Andreeva:** Validation, Visualization, Writing - original draft, Writing - review & editing. **Chuanren Ye:** Validation, Visualization, Writing - original draft, Writing - review & editing. **Kostya S. Novoselov:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing - original draft, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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