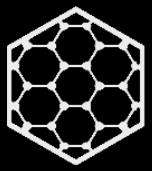


Graphene Characterization Survey

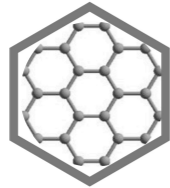
Survey of Graphene Materials Used in Various Applications and the Most Relevant Material Characteristics






The Graphene Council

About



The Graphene Council

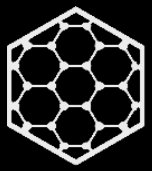
The Graphene Council is;

- 
The largest community in the world for graphene researchers, developers, producers and other stakeholders.
- 
Formal members of the ISO/ANSI/IEC Graphene Standards development working groups.
- 
Produce original information, content and reports on the state of the graphene industry and takes a lead in facilitating commercialization through education and networking.

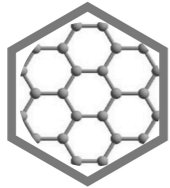
The screenshot shows the website interface with the following elements:

- Header:** "The Graphene Council" logo and name, with navigation links: "About -", "Join", "News -", "Events", "Journal", "Standards -", "Directory", "Resources".
- Utility:** "Contact Us", "Sign In", "Register", a search bar, and social media icons for Twitter and LinkedIn.
- Main Banner:** "Graphene Market Report" featuring a book cover, a quote from James Baker, and his photo.
- About Us Section:** Text describing the council as the largest community, listing industries impacted (Composites, Sensors, Electronics, etc.), and a photo of Terrance Barkan, Executive Director.
- Sign In Section:** "Sign In securely" button and a link for "Haven't registered yet?".
- Resources Section:** "2017 Bulk Graphene Market Report" and "2016 Global Graphene Survey Report" with brief descriptions.
- Latest News Section:** A list of news items with dates, such as "Concrete Graphene Applications, Literally!" and "Graphene Fire Retardant Demonstration".
- Calendar Section:** A list of upcoming events, including "Graphene and Composite Materials Webinar" and "Global Composites Conference - Las Vegas".





Graphene News and Information



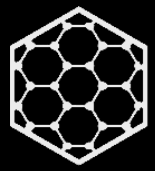
The Graphene Council

- Weekly graphene intelligence newsletter.
- Graphene Technology peer reviewed journal published by Springer Nature.
- Graphene Bulk Pricing and other market oriented reports.
- The most in-depth source of information on graphene commercialization

Visit www.TheGrapheneCouncil.org

The screenshot shows the homepage of The Graphene Council website. At the top is the logo and navigation menu with links: About, Join, News, Events, Journal, Standards, Directory, Resources. Below the menu is a banner for 'Graphene Council Corporate Members' featuring logos for Applied Graphene Materials, talga, haydale, imagine, CEALTECH, SPIE, OVA, first graphite, williambythe, and nanoXPLORE. The main content area is divided into sections: 'About Us' with a description of the council and a photo of Terrance Barkan, Executive Director; 'Sign In' with a login button and a link for new users; 'Resources' featuring a '2017 Bulk Graphene Market Report'; 'Latest News' with a list of recent articles; and 'Calendar' with a list of upcoming events.

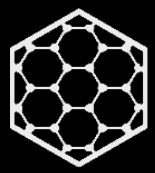




Forms of Graphene

Types of Graphene Materials Used in Target Applications

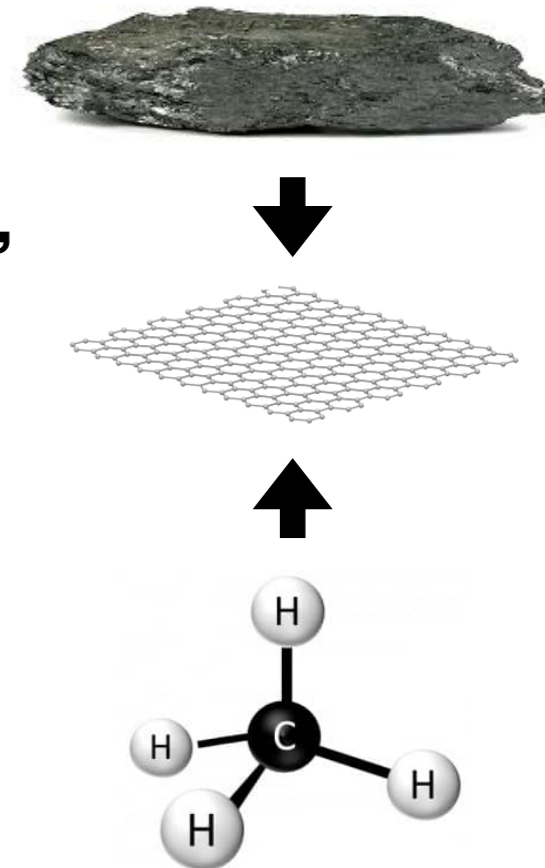


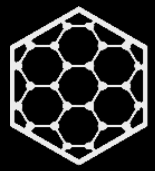


Forms of Graphene

Production

- Graphene production methods can be classified broadly as “Top Down” and “Bottom Up”.
- “Top Down” methods start with a feedstock material such as graphite and through various methods (physical, electrical, chemical, etc.) exfoliate individual layers of carbon.
- “Bottom Up” methods start with a carbon feedstock such as methane gas that under controlled conditions (such as Chemical Vapor Deposition-CVD) is deposited on a substrate material (such as copper) in single or multiple layers.



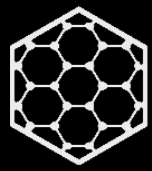


Forms of Graphene

- ❖ A wide range of commercially available materials are referred to as “graphene”
- ❖ “ISO/TS 80004-13:2017(en) Nanotechnologies – Vocabulary – Part 13: Graphene and related two-dimensional (2D) materials” recognizes material up to and including 10 carbon layers as “graphene”
- ❖ IEC Blank Material Specification for Graphene
- ❖ ISO TC229 / IEC TC113 Graphene Characterization Matrix

Number of Carbon Layers	Description
1	CVD, Mono-layer or “Pristine” Graphene
1 - 3	Very Few Layer Graphene (vFLG)
2 - 5	Few Layer Graphene (FLG)
2 - 10	Multi-Layer Graphene (MLG)
> 10	Exfoliated graphite, “Graphene nanoplatelets” (GNP) or “nano graphite”

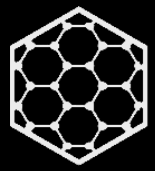




Graphene Characterization

- ❖ Besides the number of carbon layers, additional characteristics define the material.
- ❖ Graphene Oxide (GO) - a compound of carbon, oxygen and hydrogen (typically approx. 65% carbon / 35% oxygen by weight).
- ❖ Reduced Graphene Oxide (rGO) - Graphene Oxide in which removes much of the oxygen content resulting in approximately 95% carbon by weight.
- ❖ Graphene Powder, Solution or Paste - Graphene material can be prepared in various physical forms including as a dry (usually black) powder, in solution (e.g. water or alcohol) or in a paste form (often as a dull reddish brown color).
- ❖ Graphene Nano Platelets (GNPs) - GNPs typically have thickness of between 1 nm to 3 nm and lateral dimensions ranging from approximately 100 nm to 100 μm .
- ❖ Functionalized Graphene - Chemical functionalization (adding specific elements to the surface of the graphene) is important in many applications where untreated graphene would be difficult or impossible to work with.



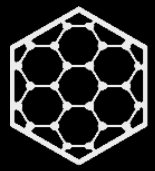


Graphene “Quality”

There is widespread confusion about the definition of “Quality Graphene”.

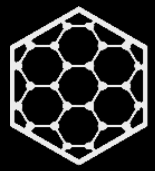
- ❖ **Material that is not suitable for one application may be ideal for another.**
- ❖ **Graphene “defects” may actually enhance the efficacy of the material for a particular application.**
- ❖ **There is no such thing as a reference material for graphene at this time.**
- ❖ **Because load factors can be quite low, the price of the material is not the most significant factor when selecting source material.**
- ❖ **The primary factor is in selecting a trusted, competent and consistent supplier of the material that understands your application areas.**





Review of Graphene Material Data Sheets from Commercially Available Products





Lack of Transparency

More than 60 different graphene material data sheets were reviewed.

More than 45 different discrete characteristics or pieces of material information were listed one or more times for one or more of the products.

Not a single material characteristic was shared across every product.

The most commonly reported characteristics were provided for fewer than 75% of the products we analyzed.

General Product Description			
		Min	Max Target
%Carbon (LOI)		99	100 <input type="checkbox"/>
Surface Area m2/g		335	365 <input type="checkbox"/>
Type: Synthetic			
Typical Analysis		Test Methods	
<i>(U.S. Standard Test Sieves)</i>			
%Carbon (LOI)	99.67	%Carbon	E4-1
%Ash	0.33	%moisture	E4-7
%Moisture	1.93	Surface Area	E3-9
Surface Area m2/g	353.19		

Properties

Chemical name: Multi Layer Graphene (MLG)
 Form: Dispersion
 Solvent: Color: Black
 Odor: Odorless
 pH: ~7.0
 Composition: C (100 %),
 Layers (N): $4 \geq 6$
 Flake size: $\sim 0.5 \pm 0.2 \mu\text{m}$

Characterization

The Raman plot shows Intensity (a.u.) on the y-axis and Raman shift (cm⁻¹) on the x-axis, ranging from 1000 to 3000. A sharp G band peak is visible at approximately 1580 cm⁻¹, and a broader 2D band peak is visible at approximately 2700 cm⁻¹. The 2D band is significantly more intense than the G band, indicating a high degree of graphitization.

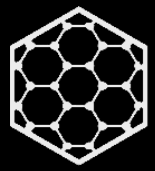
Layers: ~10 layers thick (90% of material)

Composition: Oxygen: 5-10% (dehydrated sample)

Non-oxygen composition:

Carbon:	>99.8%
Silicon:	< 40 ppm
Phosphorous:	< 200 ppm
Sulphur:	< 60 ppm
Potassium:	< 5 ppm
Calcium:	< 30 ppm
Chromium:	< 125 ppm
Manganese:	< 10 ppm
Iron:	< 900 ppm
Nickel:	< 20 ppm
Copper:	< 5 ppm
Zinc:	< 2 ppm



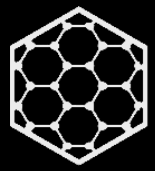


Graphene Data Sheets

List of Characteristics on Material Specification Sheets (Alphabetical order)

Ash by WT%	Metallic Content ppm	Residual Acid Content
Average Lateral Dimensions (x&y) um	Moisture % (Powders Only)	Salts ppm
Average Through Plane Dimension (z) nm	Nickel ppm	Sheet Resistance Ohm/
Bulk resistivity	Nitrogen by WT%	Shelf Life
Calcium ppm	Number of Layers	Silicon ppm
Carbon %	Odor	Solids %
Chromium ppm	Oxygen by WT%	Solvent
Color	Particle Size Distribution um	Specific Surface Area m2/g
Combustion point in Degrees C	pH	Sulphur ppm
Copper ppm	Phosphorous ppm	Tap Density g/cm3
Electrical Conductance	Porosity nm	Tensile Modulus
Functional Groups AT%	Potassium ppm	Thermal Conductivity (watts/meter-K)
Hydrogen by WT%	Primary Sheet Aspect ratio	True/Bulk Density g/cm3
IMAGES (Microscopy)	Purity Level	Visual
Iron ppm	RAMAN Scans (D, G and 2D Bands)	Zinc ppm
Manganese ppm	Relative Gravity g/cc	





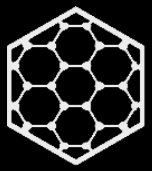
Graphene Characterization Survey

The Graphene Council received 373 replies to our graphene characterization survey. The objectives of the survey included to;

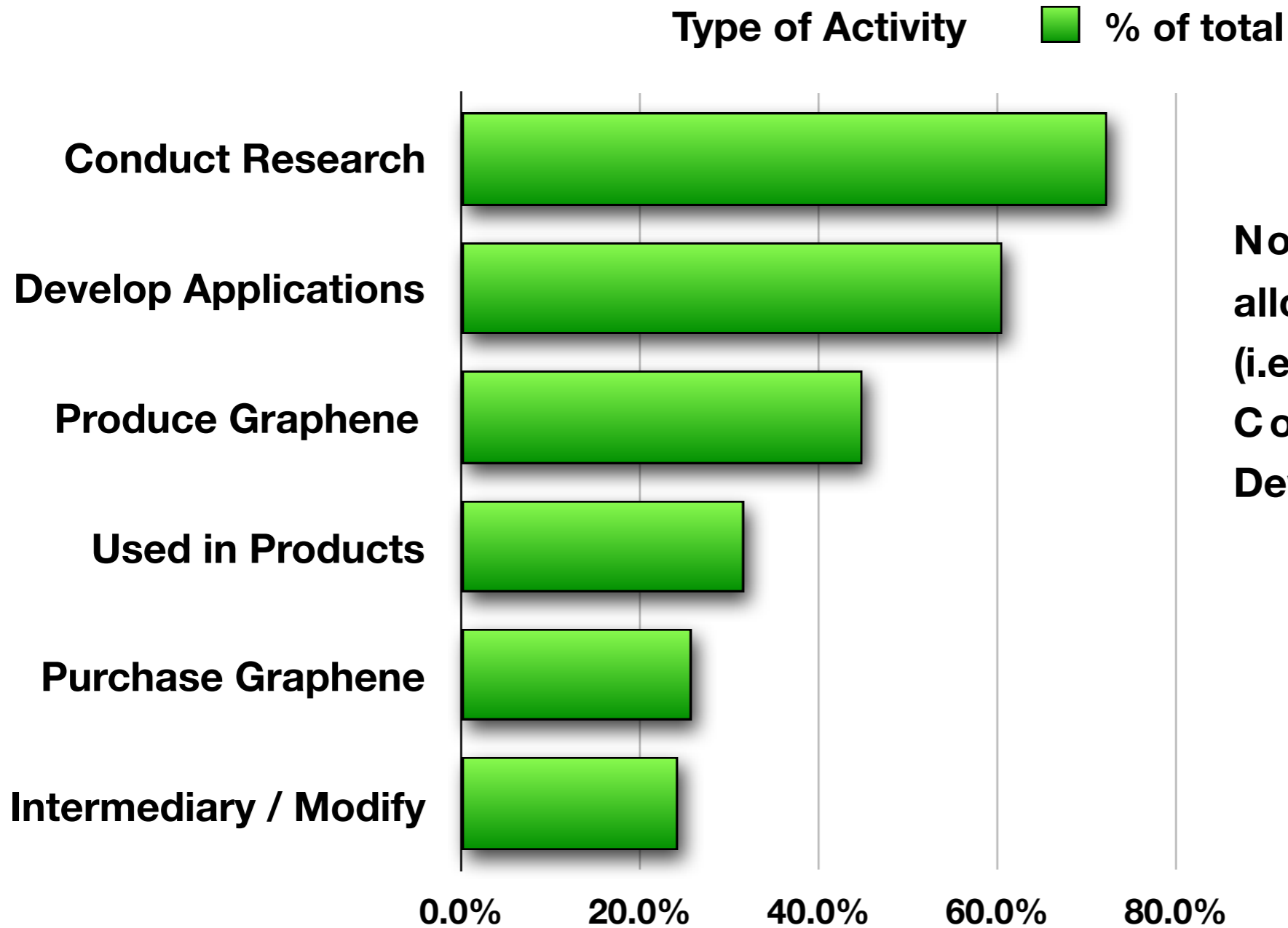
- 1. Understand what types of graphene are being used,**
- 2. For which types or categories of applications and,**
- 3. Which material characteristics are of highest importance or value.**

In addition, we asked respondents to declare if they were a graphene producer, researcher, application developer or end user.



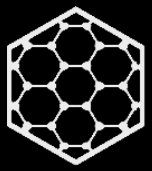


Graphene Characterization Survey



Note: Respondents were allowed multiple responses (i.e. they may be a Producer, Conduct Research and Develop Applications)

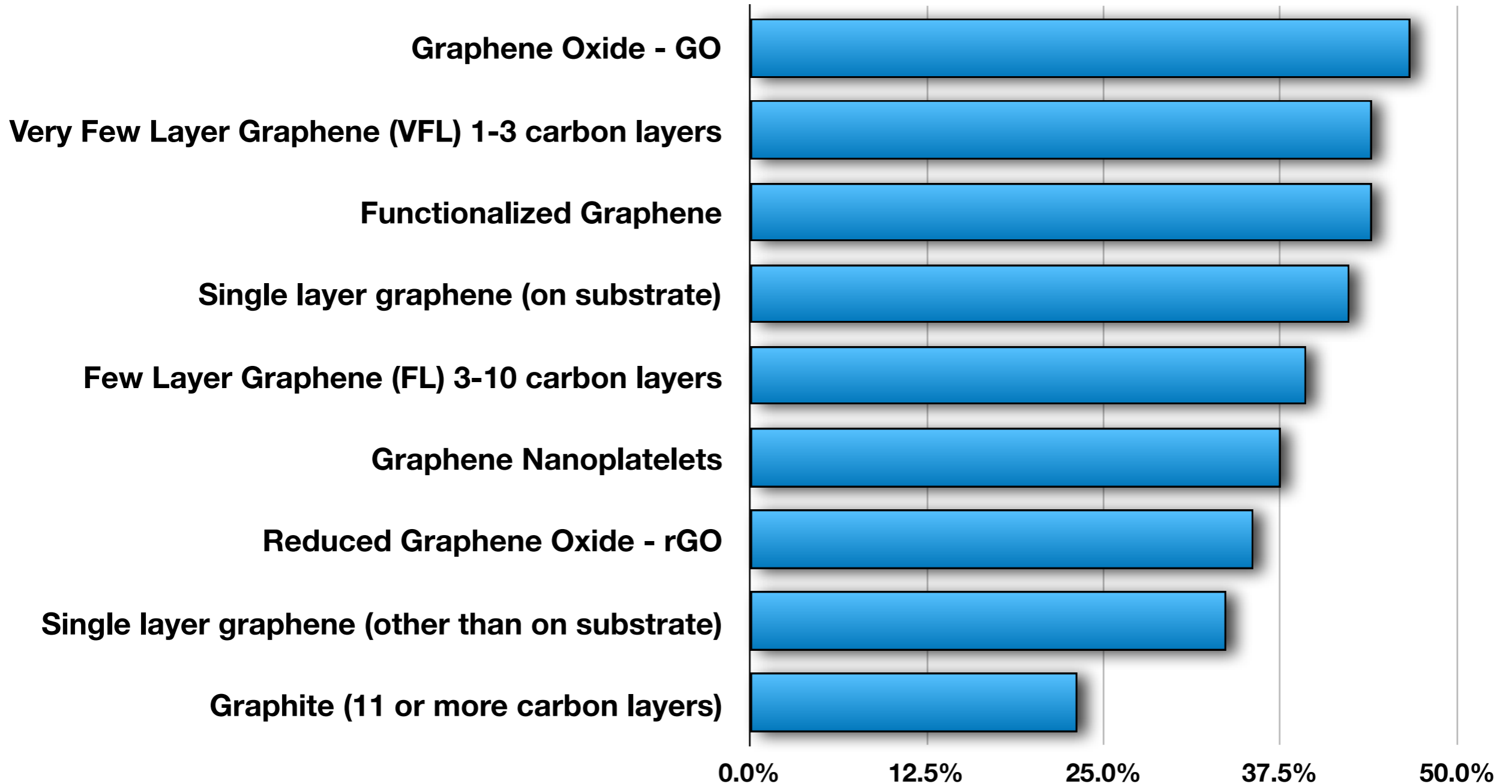


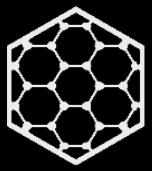


Graphene Characterization Survey

Type of Material Used

■ % of Total

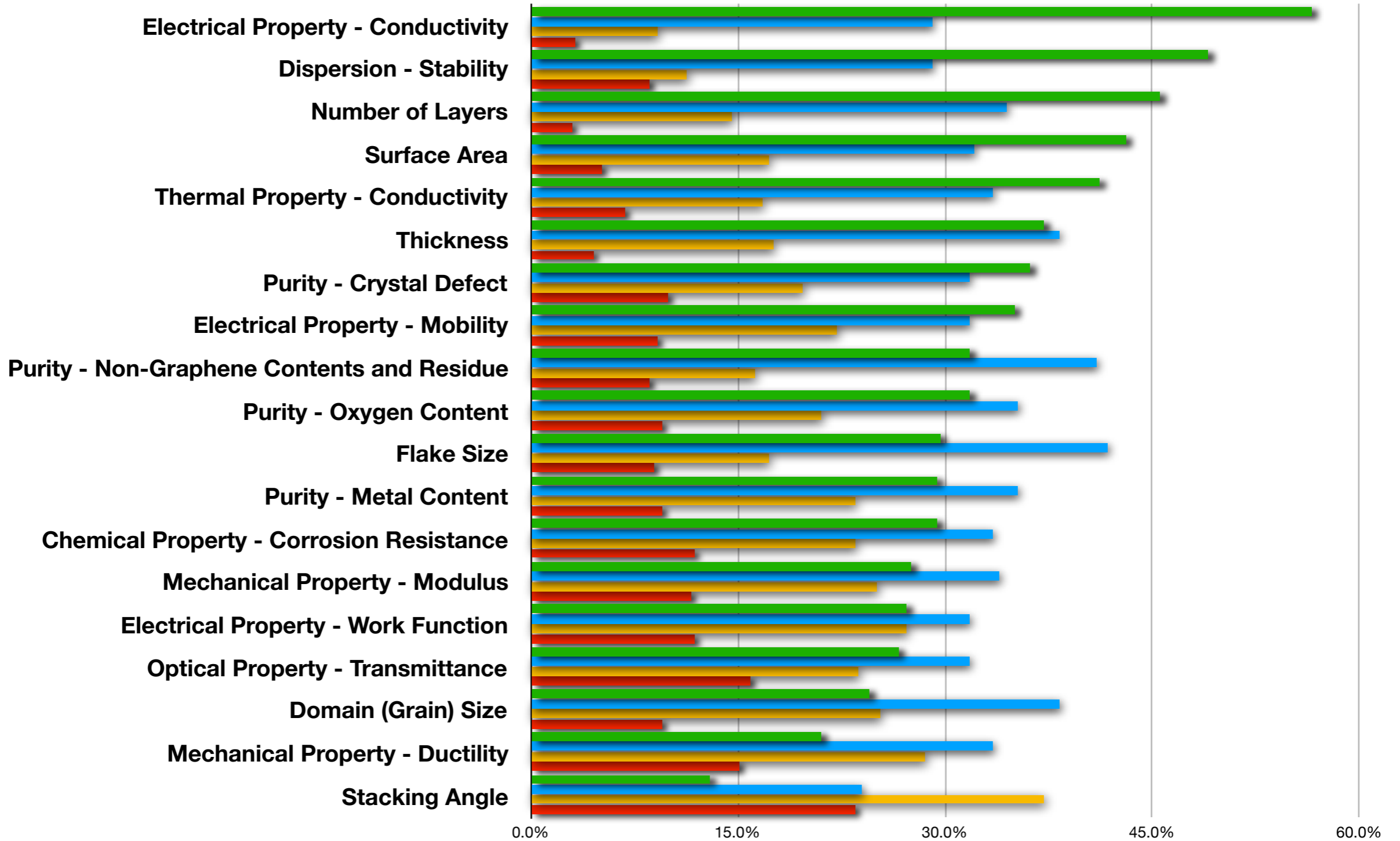


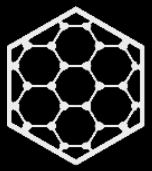


Graphene Characterization Survey

Prioritization of Characteristics

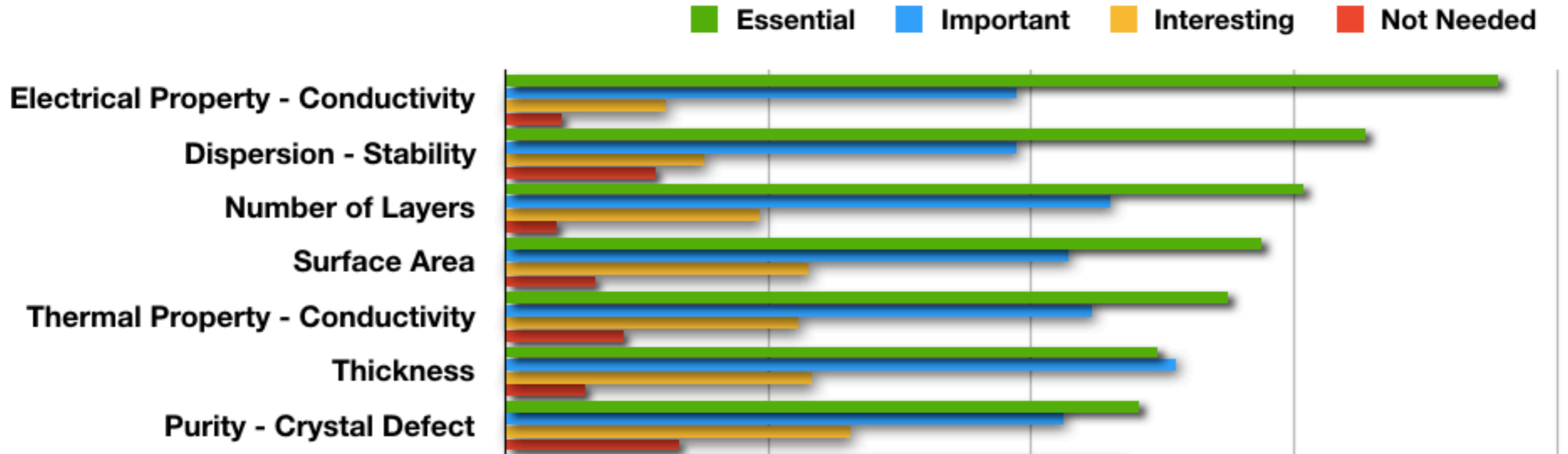
■ Essential ■ Important ■ Interesting ■ Not Needed

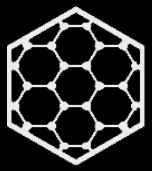




Graphene Characterization Survey

Prioritization of Characteristics



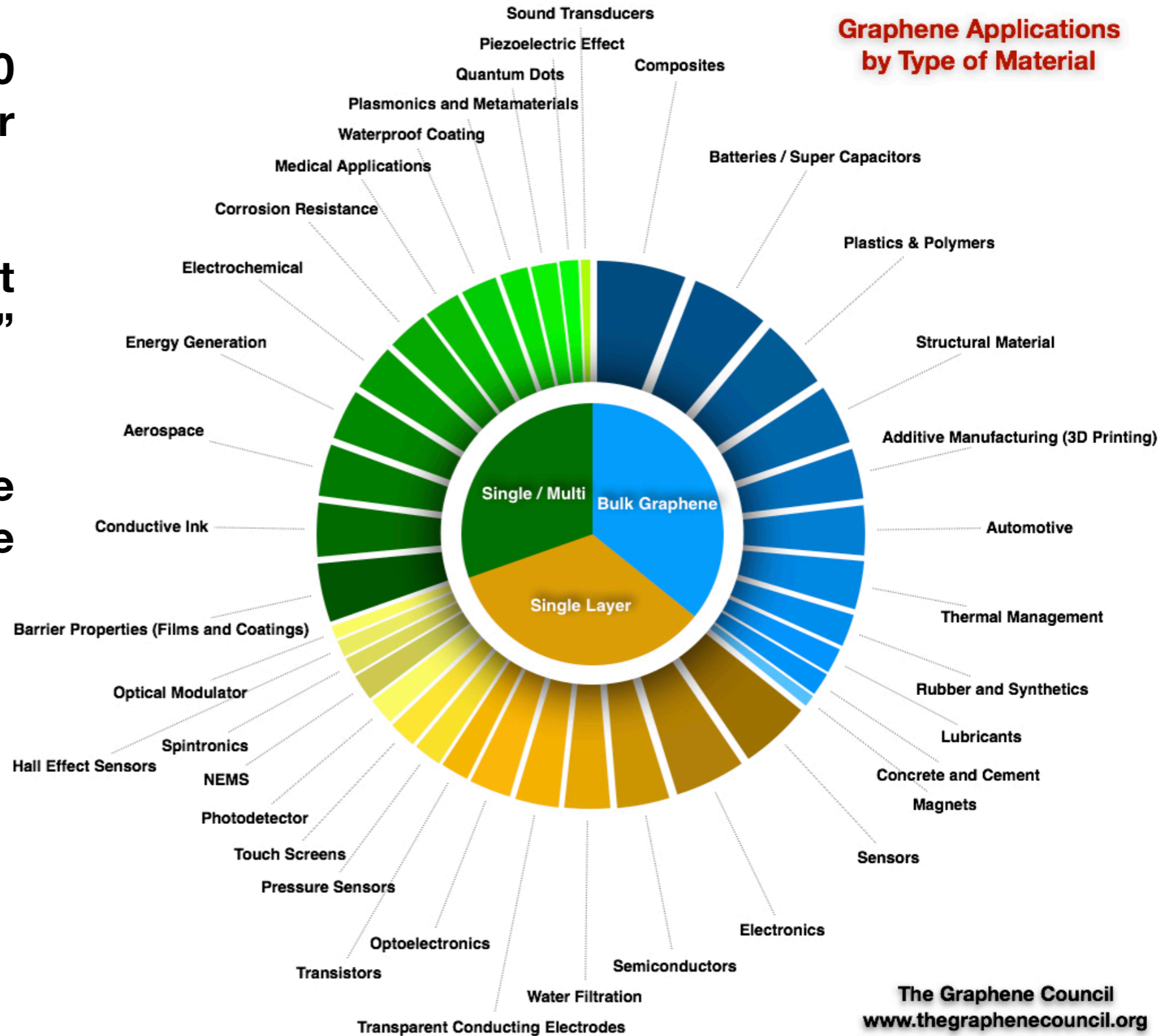


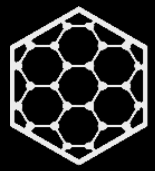
Graphene Applications

There are more than 40 major applications areas for graphene.

‘Composites’ are the largest application area for “bulk” graphene.

Sensors and electronics are key applications for single layer graphene.

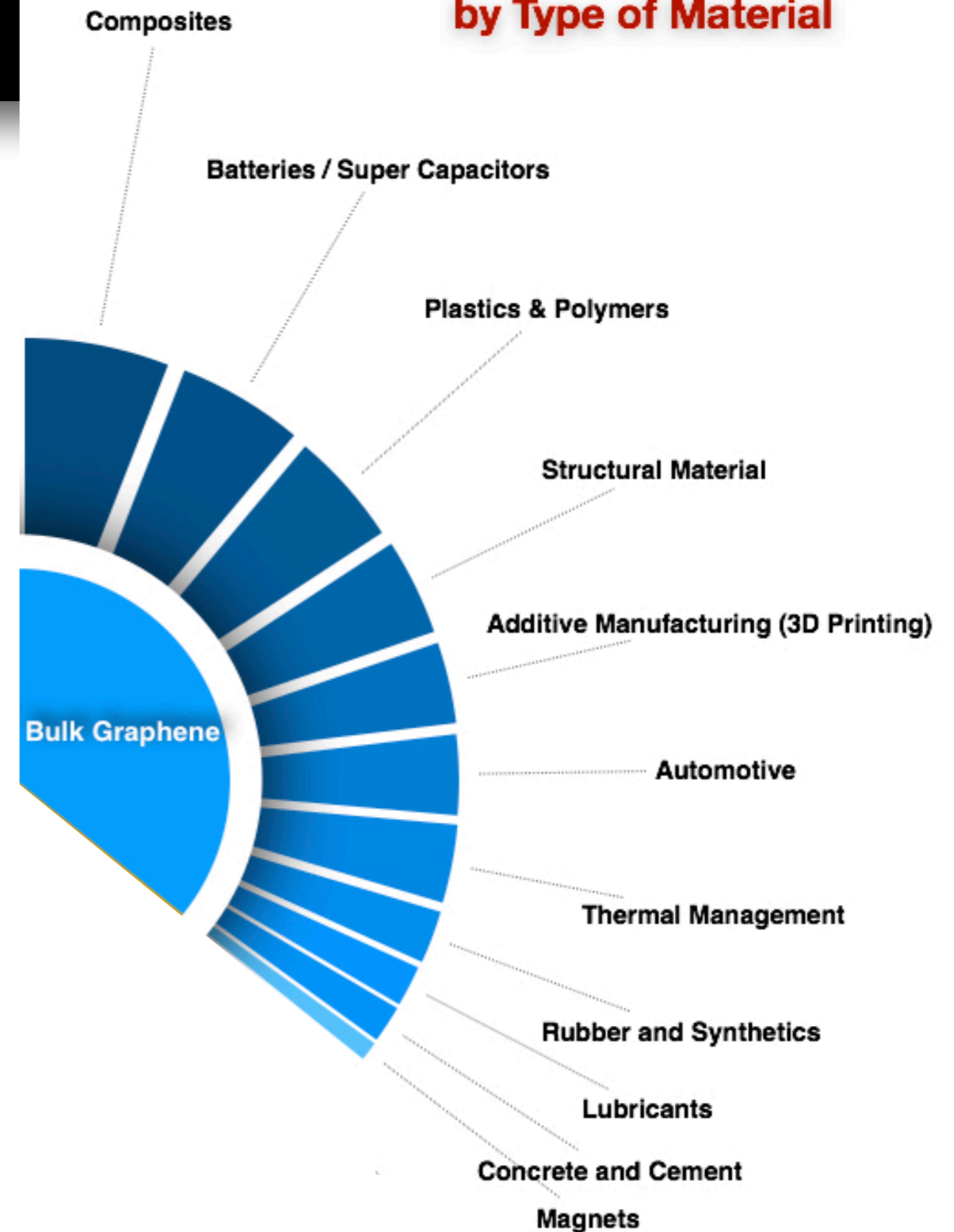


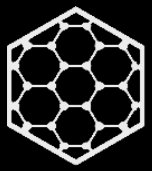


Applications

“Bulk Graphene” includes Graphene Oxides, GNP’s and multi-layer graphene materials usually in the form of powders, suspensions or incorporated in ‘master batch’ host materials

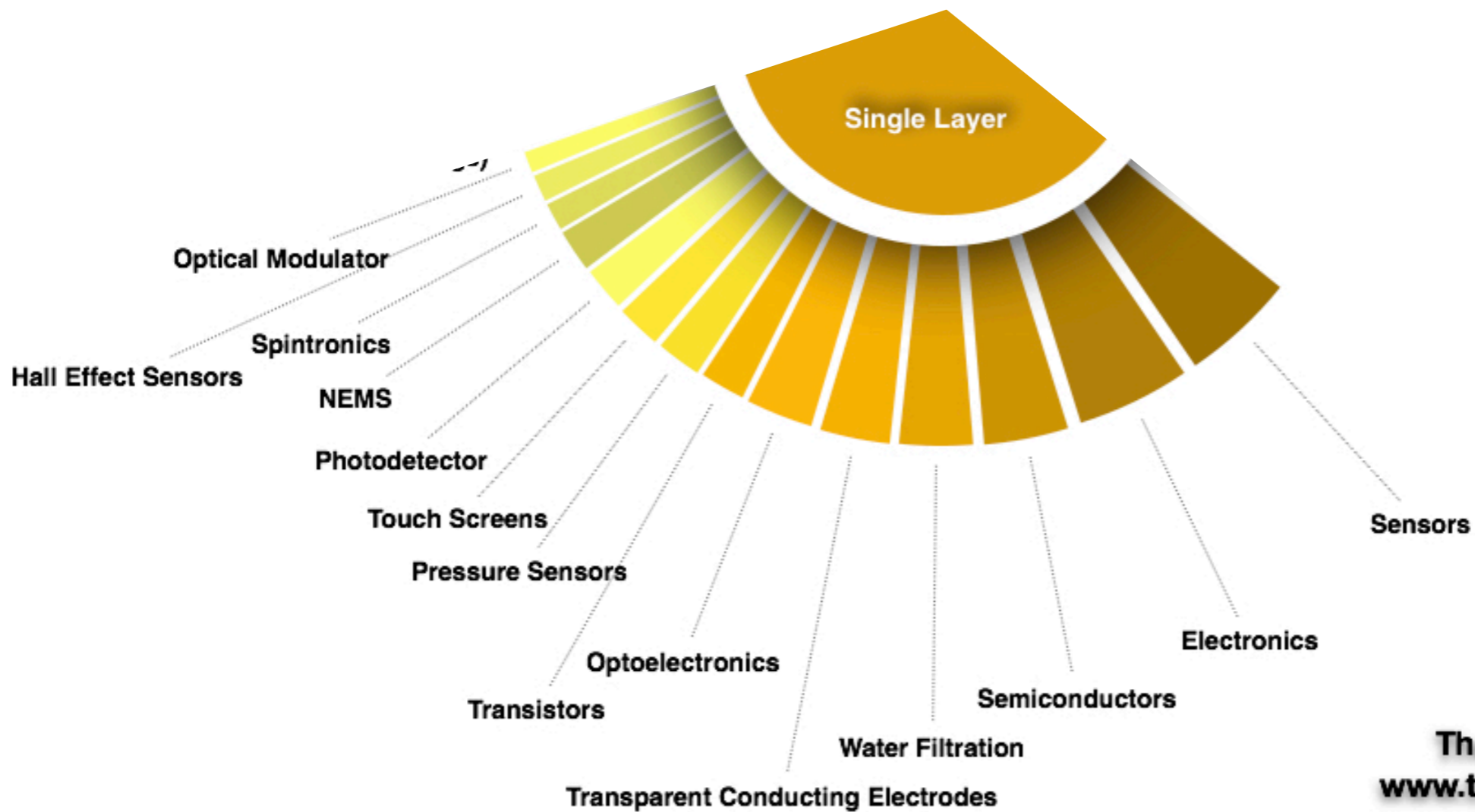
Graphene Applications by Type of Material





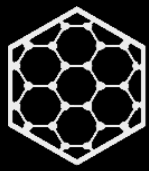
Graphene Applications

'Single Layer' is almost exclusively CVD or Epitaxial Graphene.

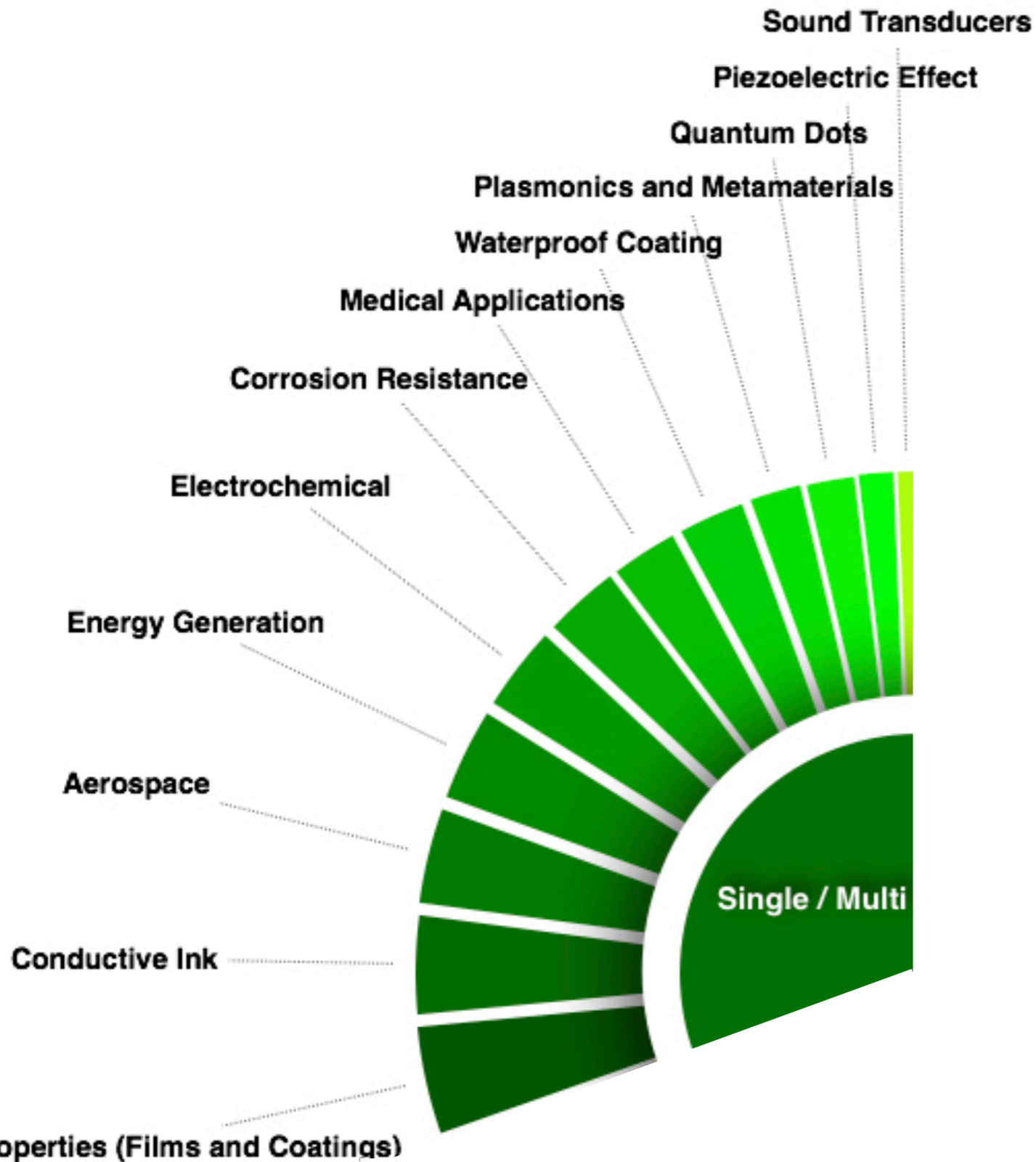


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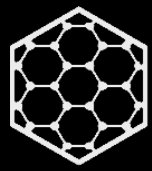




Graphene Applications



These applications may use a combination of types of graphene or use single layer and multilayer material interchangeably.

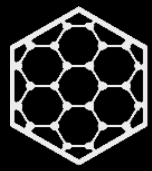


Key Characteristics & Testing Methods

Draft recommendation of a minimum set of material specifications for materials listed as “Graphene” (GO, rGO, CVD, GNPs, FLG, etc.)

Characteristic	Testing Method	Reporting Format
Number of carbon layers	RAMAN / TEM	Number of carbon layers (minimum and maximum) and distribution (e.g. 10% mono-layer, 50% 2-3 layer, 40% 4-8 layer).
Average Through Plane Dimension	RAMAN	Average Through Plane Dimension (z axis) in nm
Average lateral dimensions of the graphene flakes or nano platelets	TEM / SEM /	Average lateral dimensions (x&y axis) in μm
Specific Surface Area	BET (ISO 9277:2010)	Specific Surface Area expressed as m^2/g
Chemical analysis of functionalized graphene	XPS	Report for each element detected either by weight as a percentage of the sample or in ppm.
Carbon content	XPS	Report the amount of Carbon as a percentage of the total weight of the material.
Oxygen content	XPS	Report the amount of Oxygen as a percentage of the total weight of the material.
Raman Scan Images	RAMAN	Graphic illustration of the RAMAN Scans (D, G and 2D Bands).
Microscopy images	TEM / SEM	At least one image of the material at a sufficient magnification to visualize the structure with a scale overlaid on the image.





Graphene and Composites

Questions?

The Graphene Council

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