HORIBA Scientific

Application Note

Surface Area of Graphite

AN243

Graphite is an important and widely used material. Graphite's properties stem from its hexagonal lattice structure. It is composed of carbon atoms arranged in layers, each consisting of interconnected hexagonal rings. These layers can easily slide past one another, giving graphite its characteristic lubricity.

Introduction

The surface area of graphite is indeed of significant importance, especially in applications where the interaction between graphite and other substances or materials is crucial.

Graphite's versatility is reflected in its wide range of applications across various sectors:

- Industrial Lubricants: Thanks to its layered structure, graphite serves as an effective dry lubricant.
- Pencils and Writing Instruments: The most common association with graphite is pencils.
- Refractories and Metallurgy: Graphite's high melting point and resistance to thermal shock make it a key component in refractory materials used in the production of steel, aluminum, and other metals.
- Batteries: Lithium-ion batteries rely on graphite anodes to store and release energy efficiently.
- Electronics: Graphite is used in various electronic components, including printed circuit boards and heat sinks, due to its excellent thermal conductivity and electrical properties.

Gas Adsorption Technique

Surface area in this case is measured by a technique known as gas adsorption or physical adsorption (physisorption). Surface area is typically reported as meters squared per gram.

Adsorption takes place on the surface of a material. Gas molecules of known cross sectional area are carefully inventoried as they adhere to the surface. By knowing the number of moles of gas adsorbed and their cross section, the total surface area under test can easily be calculated.

Figure 1. An example of graphite powder.

The adsorption process occurs due to surface energy typically described as Van der Waals forces. Left alone, most materials will adsorb water or other vapors to satisfy this surface energy. Thus, to take advantage of these surface forces for measurement, the adsorbed impurities must be removed. This is accomplished through a process known as degassing, whereby a sample is placed in a holder and an inert flow of gas passes through the sample powdered bed as it is gently heated. The applied heat causes the adsorbed impurities to break free of the surface of the material and the flow of gas sweeps them away.

Cleaned in such a way, the sample holder may now be placed on an analysis station where it is cooled (typically to liquid nitrogen temperatures) in a cryogen Dewar as a gas mixture (typically nitrogen in a carrier gas of helium) flows across the surface. As the sample and gas cool, nitrogen molecules in the flowing gas mixture lose energy and are adsorbed onto the sample surface.

Again, monitoring the number of moles of gas adsorbed as a function of gas concentration allows us to calculate several adsorption data points. For more rapid analysis, a single point may be collected. The most common calculation method applied to derive specific surface area is the Brunauer, Emmett and Teller (BET) method.

Experimental

A commercially available graphite powder sample was sent to the HORIBA Instruments Incorporated applications lab in Irvine, California for analysis. The sample was analyzed using the new SA-9650 surface area analyzer. Three separate aliquots each weighing approximately 0.15 g were placed in sample tubes. Degassing was performed on the 3 integrated degas stations of the SA-9650 at 300˚C for 2 hours. Samples were then transferred to the 3 analysis stations and separate analyses were performed for single point and multi point surface area. Each type of test was repeated 3 times. The results are shown in the table below.

Single Point*

	Channel 1	Channel 2	Channel 3	Average of 3 Channels	CoV (%)
Test 1	5.55	5.8	5.74	5.70	1.87
Test 2	5.60	5.92	5.78	5.77	2.27
Test 3	5.65	5.96	5.78	5.80	2.19
Average of 3 Tests	5.6	5.9	5.8		
CoV (%)	0.73	1.15	0.33		

Multi-Point*

	Channel 1	Channel 2	Channel 3	Average of 3 Channels	CoV (%)
Test 1	5.87	6.09	5.94	5.97	1.54
Test 2	5.55	6.24	5.99	5.93	4.81
Test 3	5.65	6.33	6.08	6.02	4.66
Average of 3 Tests	5.7	6.2	6.0		
CoV (%)	2.35	1.59	0.96		

*The difference between single point and multi point analyses are linked to assumptions made in the single point calculation. Multi-point results are typically more accurate. However, in terms of repeatability and reproducibility, either measurement is quite robust and single point measurements are extremely fast – supporting high throughput, production, or quality control environments.

Conclusion

The HORIBA SA-9650 surface area analyzer proved to be an ideal instrument for measuring the surface area of graphite both by single and multi-point analysis. The analyses were fast, repeatable and the instrument is robust. The methodology described in this document should be useful as a guide to using the SA-9650 for graphite or other powdered samples.