Australia's National
Science Agency

From minerals to materials

Supplementary report: Graphite

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CSIRO acknowledges the Traditional Owners of the lands that we live and work on across Australia and pays its respect to Elders past and present.

The project team would like to acknowledge the contributions of all stakeholders that provided input to this project from industry, government and academia. Appendix A of the main report 'From minerals to materials: Assessment of Australia's critical mineral mid-stream processing capabilities' includes a complete list of the organisations that provided input into to this project.

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Contents

Glossary

Abbreviations

ALD	Atomic layer deposition
CAM	Cathode active material
C-SPG	Coated spherical purified graphite
CVD	Chemical vapour deposition
DRC	Democratic Republic of Congo
EV	Electric vehicle
FBICRC	Future Battery Industries Cooperative Research Centre
GWh	Gigawatt-hours
HF	Hydrofluoric acid
HPA	High purity alumina
IEA	International Energy Agency
IP	Intellectual property
LCE	Lithium carbonate equivalent
LIB	Lithium-ion battery
LMB	Lithium metal battery
LTO	Lithium titanate oxide
NaOH	Sodium hydroxide
NMC	Nickel manganese cobalt oxide
OEM	Original equipment manufacturer
pCAM	Precursor cathode active material
RD&D	Research, development and demonstration
SEI	Solid electrolyte interface
SPG	Spherical purified graphite

1 Executive summary

The global energy transition and demand for lithium-ion batteries (LIBs) is driving substantial growth in the graphite market, both natural and synthetic. It is also driving growth in the production of silicon-graphite composite anode materials which are pushing the limits of lithium battery performance. The level of demand and significant concentration of production in China[1] is driving changes across global supply chains. Companies with deposits outside of China are seeking to vertically integrate by expanding their services and producing value-added graphite products.[2] This shift is supported by policies and legislation from the US and EU, strong market demand from automobile manufacturers,[3] and recent reductions in graphite exports from China.[4] The growing demand for graphite, alongside strategic diversification initiatives and the pursuit of sustainable supply chains, presents Australia with an opportunity to leverage its deposits and move up the supply chain.

Although Australia has no commercial scale production of graphite materials, several companies have conducted tests and pilots, and plan to vertically integrate from their natural graphite deposits. There are several opportunities for research, development and demonstration (RD&D) across the graphite supply chain to support the growth of an onshore industry, including supporting the implementation of mature technologies from overseas at commercial scale; demonstrating Australian IP at scale; and accelerating emerging technologies to grow Australian IP (Figure 1 and Figure 2).

This supplementary report is part of the report series *From minerals to materials: Assessment of Australia's critical mineral mid-stream processing capabilities*. The series adds to existing Australian and international literature on critical minerals and renewable energy technologies by providing a detailed picture into mid-stream processing, key areas for global risk reduction and capability development to support the energy transition in Australia.

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[1] Nouveau Monde Graphite (2021) Graphite 101: Powering the Clean Energy Transition. <https://nmg.com/wp-content/uploads/2021/06/NMG-Graphite-101.pdf>

[2] Loh J (2022) Powering Australia: how CSIRO is helping industry meet demand for graphite. CSIRO. <https://www.csiro.au/en/work-with-us/industries/mining-resources/resourceful-magazine/issue-27/graphite-processing-for-batteries>

[3] Onstad E (2023) Analysis: Auto firms race to secure non-Chinese graphite for EVs as shortages loom. Reuters.

[4] Bloomberg News (2024) Chinese Exports of Battery Material Graphite Plunge on Controls. <https://www.bloomberg.com/news/articles/2024-01-22/chinese-exports-of-battery-material-graphite-plunge-on-controls>

Figure 1: Framework for assessing research, development and demonstration (RD&D) and international engagement actions.

Opportunity area	Establish new capability in emerging technologies	Accelerate emerging technologies and grow Australian IP	Pilot and scale up Australian IP	Support commercial deployment of mature technologies
RD&D actions	Build capability in emerging technology areas via fundamental and applied research projects.	Leverage Australia's strengths to progress technologies beyond the lab and grow Australian IP.	Deploy Australian IP in pilot-scale and commercial-scale demonstrations.	Support the deployment of mature technologies domestically at commercial scale, through commercial testing and validation, and cross-cutting RD&D.
International engagement actions	Engage with research institutions on capability building and knowledge sharing (e.g. joint research programs).	Partner with overseas industry, research or government on mutually beneficial sustained technology development efforts (e.g. co-funded or joint projects).	Engage with upstream offtakers to de-risk and finance pilot projects. Alternatively, demonstrate Australian technologies overseas.	Engage on commercial arrangements e.g. international technology providers, license overseas patents, attract foreign direct investment, and secure offtake agreements.

For a full description and methodology of this framework, refer to the main report *From minerals to materials: Assessment of Australia's critical mineral mid-stream processing capabilities.*

IP, intellectual property.

Micronisation and spheronisation

Micronisation and spheronisation technology is mature globally and off the-shelf equipment is accessible to Australian industry. Continued collaborations with equipment suppliers, and strong engagement with overseas battery original equipment manufacturers (OEMs) will be key to growing onshore production.

Despite the use of overseas technology, there is a role for Australian RD&D to optimise the operation of commercial equipment, with the goal of improving yields and efficiency. Although this doesn't fall under the purview of traditional RD&D activities, it could be a crucial for enhancing the performance of the domestic industry, especially considering the variability in equipment and ore characteristics.

HF acid-free purification

Diversifying graphite purification outside of China will require the development hydrofluoric (HF) acid-free methods due to its high risks, and Australia is well positioned to do so with strong RD&D and patent output to support piloting and scale-up.

Australia has developed patents across several alternatives to HF-acid, such as chlorination roasting and alkaline roasting or leaching pathways. Continued R&D efforts are required to scale up alternative purification pathways (that are HF-acid-free) beyond the laboratory and pilot scales. Given Australian companies hold graphite deposits in Australia and internationally, Australian purification technologies could support value-adding operations onshore and offshore, unlocking greater global diversification.

Coating processes

There are significant barriers to entering the C-SPG market because coating technology is highly subject to trade secrets; however, there is an opportunity to develop formulations and coating methods in collaboration with international partners.

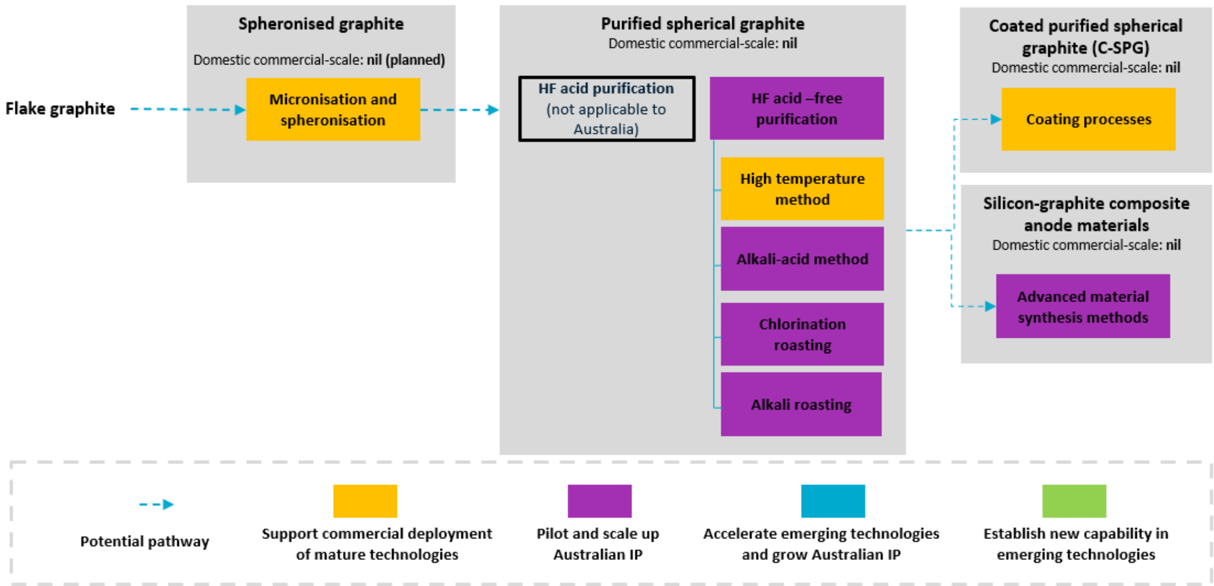
Coating technologies are mature globally, and Australia can leverage its strong RD&D capabilities in advanced materials (e.g. thin films), to support commercial production of C-SPG onshore. C-SPG attracts a significantly high price compared to uncoated material. Collaborations with global battery cell manufacturers will be essential to capturing this value through the co-development of coating formulas tailored to specific manufacturers products.

Graphite composite materials (advanced material synthesis)

Australia has several active companies and research institutions working towards next generation silicon-graphite composite anodes. There is an opportunity to demonstrate Australian silicon-graphite anode materials in battery applications and pilot their production at scale, and to continue to develop novel compositions with increased performance.

Increasing proportions of silicon are being used in commercial LIBs and are achieving breakthroughs in performance, representing a significant opportunity for Australian technology developers. Demonstrating Australian anode materials and strong engagement with international LIB manufacturers will be required for Australian technology to capture anode market share.

Figure 2: Australian RD&D opportunities across graphite processing technologies.



Note: This diagram represents a simplified summary of research, development and demonstration (RD&D) actions and international engagement actions for Australia. However, some technologies and their variants cut across a range of maturity levels, therefore warranting multiple actions.

C-SPG, coated spherical purified graphite; HF, hydrofluoric acid; IP, intellectual property.

2 Objectives and scope

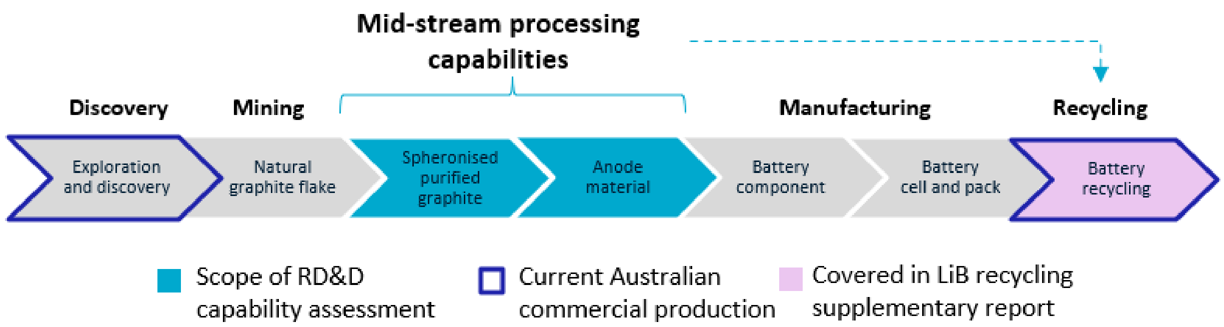
This supplementary report will focus on Australia's key graphite value-adding priorities, namely the production of anode materials (Figure 3). Given Australia's graphite deposits, this section will focus on processing of natural graphite.

This supplementary report aims to address several objectives:

- To introduce the key current and emerging technologies that underpin the processing of graphite ores into the production of anode active materials.
- To present the level of IP and research activity occurring in Australia and globally, for each emerging and mature technology area.
- To identify key challenges and opportunities for Australia to build domestic IP and collaborate with international partners, based on technological maturity, IP trends and research activity.

The purpose of this analysis is to guide and inform government, industry and research decision-making with respect to research, development and demonstration (RD&D) investment and collaboration efforts across critical minerals and renewable energy technology supply chain activity.

Figure 3: Scope of graphite supplementary report and current commercial production in Australia.



LIB, lithium-ion battery.

3 RD&D challenges and opportunities

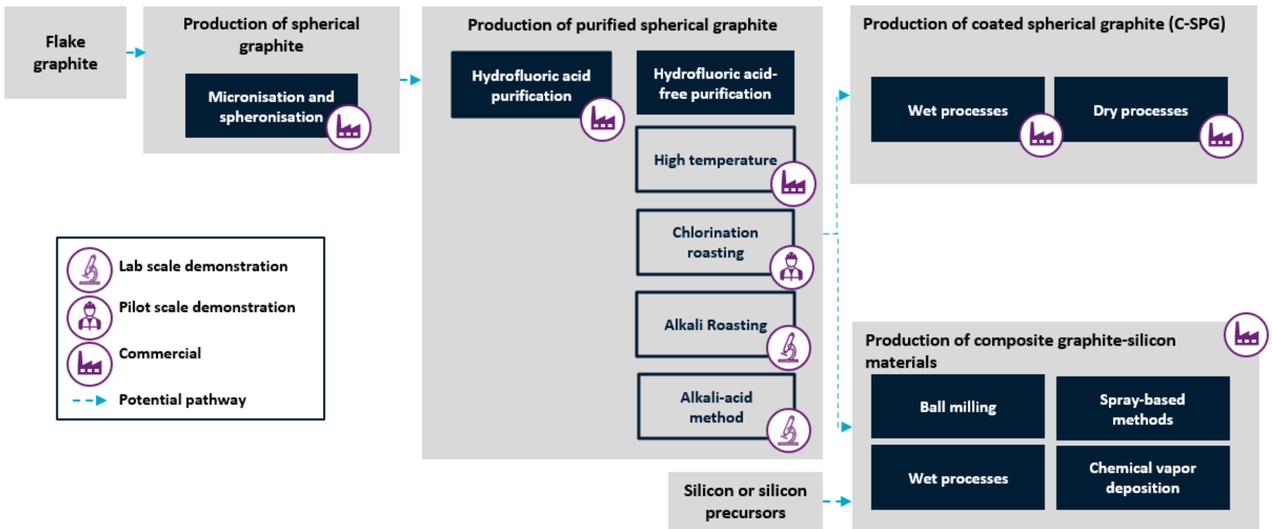
Current lithium-ion batteries (LIBs) employ anodes that are primarily composed of graphite, sourced from natural graphite deposits or made from synthetic graphite. Natural graphite is categorised into amorphous, vein, or flake graphite according to the specific deposit type, with each having distinctive physical characteristics. Flake graphite is the primary focus of mining and processing activities, largely due to its suitability in energy technologies, including lithium-ion batteries.[5]

The graphite anode value chain is well established globally, underpinned by mature and commercial processing technologies. However, there are several emerging technologies being developed globally to improve sustainability, cost and performance outcomes. Furthermore, next generation battery anode components such as silicon-graphite compounds are emerging, along with the processes to make them.

This section will discuss the RD&D challenges and opportunities relating to mature and emerging technologies for the processing of natural graphite to graphite anode materials.

- Section 3.1 will cover technologies used to shape and round natural graphite, including micronisation and spheronisation.
- Section 3.1.2 will cover the methods used to purify spherical graphite, focusing on various hydrometallurgical and pyrometallurgical processes.
- Section 3.2.3 will focus on the application of a carbon coating onto the spheronised purified graphite, encompassing both wet and dry processes.
- Section 3.3.4 will cover the synthesis of silicon-graphite composite materials for anodes.

Figure 4: Taxonomy of graphite processing technologies for the lithium-ion battery supply chain.



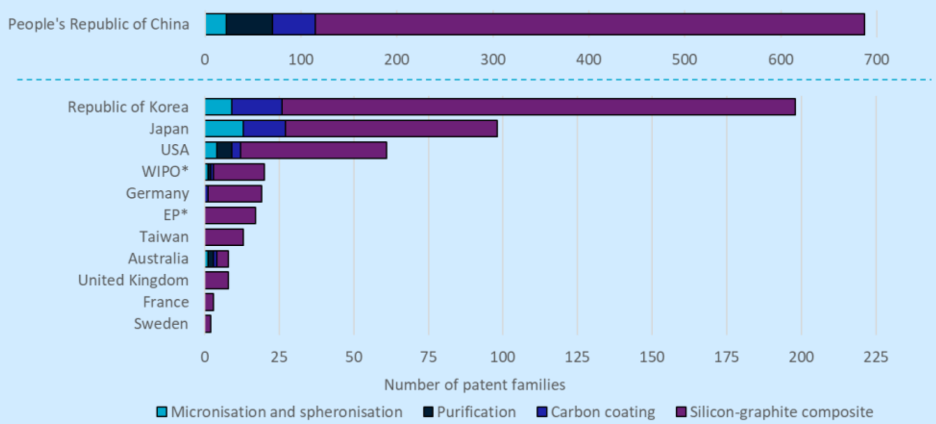
C-SPG, coated spherical graphite.

[5] Ritoe A, Patrahau I, Rademaker M (2022) Graphite: Supply chain challenges & recommendations for a critical mineral. The Hague Centre for Strategic Studies.

Global R&D and commercialisation snapshot

Production of graphite anode materials

Figure 5: Patent output in graphite processing and the production of silicon-graphite compound materials from 2007 to 2022, by country.

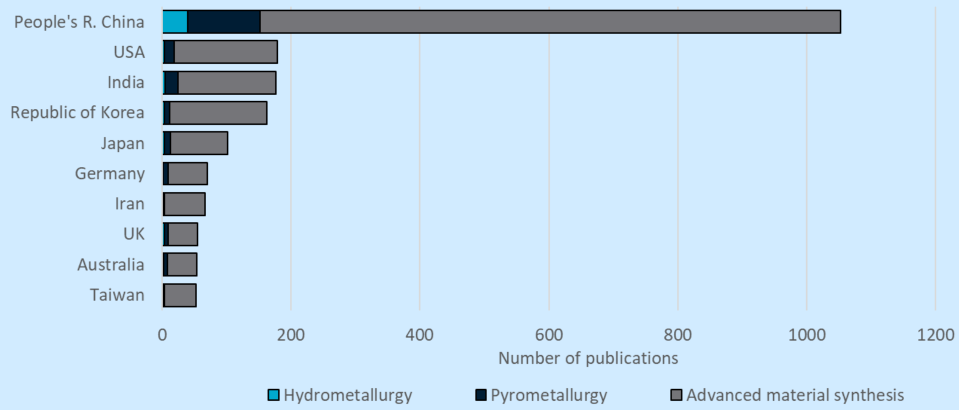


*Applications filed under an entity other than a country.

People's R. China, People's Republic of China; WIPO, World Intellectual Property Organisation; EP, European Patent Office.

Figure 5 presents global patent activity in graphite processing technologies by country, between 2007 and 2022. China had the highest activity in all four categories evaluated, accounting for 60.4% of patent filings overall, followed by the Republic of Korea (17.4%) and Japan (8.6%) in the top three. Australian filings represented less than 1% of global output, with activity in production of spherical graphite (micronisation and spheronisation), purification, production of coated spherical graphite (carbon coating), and silicon-graphite compound materials.

Figure 6: Research publication activity related to graphite throughout the 2007 – 2023 period, by country and processing technology.



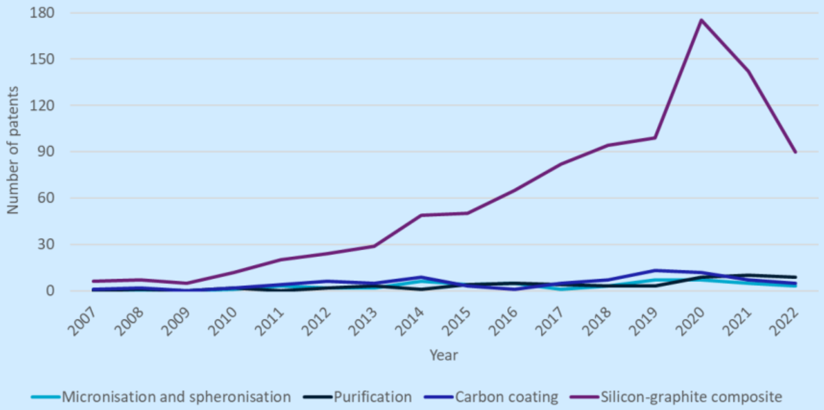
Li-B, lithium-ion battery.

Figure 6 illustrates research publications related to graphite from 2007 to 2023, by country and processing technology. Publications from China represent 40.4% of the total, with the United States following in second (6.9%), and South Korea completing the top 3 (6.6%). Australia occupies the 7th position, with 2.3% of the global output.

Synthesis of advanced materials was the area with the highest number of publications during the analysis period (73.2%), in line with the relevance of graphite in current and some next generation anode materials.

The bibliometric and patent data presented in this report is subject to limitations and has an estimated accuracy of 70% or above. For a full description of the methodology and limitations refer to the main report *From minerals to materials: Assessment of Australia's critical mineral mid-stream processing capabilities*.

Figure 7: Patent output in graphite processing and the production of silicon-graphite compound materials from 2007 to 2022, by technology.



Production of silicon-graphite compound materials had the highest patent activity, representing 83.5% of all patents identified in the analysis. The remaining three categories had lower proportions: 7.2% for the production of coated spherical graphite, 4.9% for purification, and 4.4% for micronisation and spheronisation. The filing trends over time match this distribution, with silicon-graphite compound materials seeing significant growth in activity over the past 15 years and the graphite processing technologies maintaining a mostly stable baseline (Figure 7).

Table 1: Top 10 active organisations outside of China

By research publication output	By patent output
CNRS, **France**	LG Energy Solutions, **Republic of South Korea**
IIT, **India**	Samsung SDI & Samsung Electronics, **Republic of South Korea**
UNIST, **Republic of Korea**	Shin Etsu Chemical, **Japan**
CSIR, **India**	Kyungpook National University, **Republic of South Korea**
DOE, **United States**	Resonac (former Showa Denko and Hitachi Chemical), **Japan**
NIT, **India**	SK On, **Republic of South Korea**
Helmholtz Association, **Germany**	Umicore, **Belgium**
CSIC, **Spain**	JFE Chemical, **Japan**
Inha University, **Republic of South Korea**	Wacker Chemie, **Germany**
Northwestern Polytechnical University, **United States**	Daewoo Electronic Materials, **South Korea**

The bibliometric and patent data presented in this report is subject to limitations and has an estimated accuracy of 70% or above. For a full description of the methodology and limitations refer to the main report *From minerals to materials: Assessment of Australia's critical mineral mid-stream processing capabilities*.

3.1 Production of spherical graphite

3.1.1 Micronisation and spheronisation

To prepare natural graphite for use as the anode material in lithium-ion batteries, the graphite is mechanically ground into small spherical particles. This process, called micronisation and spheronisation, reduces graphite's particle surface area (to roughly 2 to 20 microns), achieves uniformity in particle size, and increases the tap density (the volume of graphite particles that can fit into a given space).[6] These characteristics significantly enhance battery charging and discharging capabilities.

Micronisation and spheronisation is conventionally achieved by using a series of mills (as many as 25).[7] Between each milling step, the material is sorted to obtain a desired size range, which then advances to the next step. Particles not meeting the specifications are either taken through the process again or discarded as a by-product.

Alternative methods have condensed the process into a single step milling process, albeit with potentially higher initial equipment costs.[8] These technologies have several inherent advantages when compared to the conventional multi-step process, such as reducing operating and energy costs, as well as minimising the plant space required for operations.[9] One such technology, the NETZSCH GyRho Rounding Unit, can achieve yields of up to 60% whilst using 60% less energy that traditional routes.[10] Despite this, these technologies have lower commercial maturity and the RD&D challenges relating to electricity use, processing time and waste graphite material still persist.

Although milling is highly mature and off the shelf equipment is available, researchers and companies are continuously improving the technology to address the issues of high energy consumption and low yield (high waste). For example, the Alpine Particle Rounder implements a novel batch operated rounding machine as part of the process, followed by continuously operated dynamic air classifier. [11] The equipment demonstrated a 15% increase in yield and a 74% reduction in energy consumption compared to conventional methods.[12]

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6 Rapp et al. (2016) Spheroidization of Graphite As Anode Material for Li-Ion Batteries: Spheroidization Process and Material Texture and Morphology. Electrochemical Society Meeting Abstracts imlb2016 2, 503-503; Fischer et al. (2023) Impact of Spheroidization of Natural Graphite on Fast-Charging Capability of Anodes for LIB. Batteries, 9(6):305.

7 Northern Graphite (n.d.) About Spherical Graphite ("SPG"). <https://www.northerngraphite.com/_resources/media/SPG-Summary-2.pdf>; Fischer et al. (2023) Impact of Spheroidization of Natural Graphite on Fast-Charging Capability of Anodes for LIB. Batteries, 9(6):305; Engels P et al. (2022) Life cycle assessment of natural graphite production for lithium-ion battery anodes based on industrial primary data. Journal of Cleaner Production. <https://www.sciencedirect.com/science/article/pii/S0959652622001172>; Fischer S et al. (2023) Impact of Spheroidization of Natural Graphite on Fast-Charging Capability of Anodes for LIB. Batteries. <https://www.mdpi.com/2313-0105/9/6/305>

8 Fischer S et al. (2023) Impact of Spheroidization of Natural Graphite on Fast-Charging Capability of Anodes for LIB. Batteries. <https://www.mdpi.com/2313-0105/9/6/305>

9 Fischer S et al. (2023) Impact of Spheroidization of Natural Graphite on Fast-Charging Capability of Anodes for LIB. Batteries. <https://www.mdpi.com/2313-0105/9/6/305>

10 Battery Industry (2020) NETZSCH GyRho Rounding Unit: new, efficient process for the rounding of graphite. <https://batteryindustry.tech/netzsch-gyrho-rounding-unit-new-efficient-process-for-the-rounding-of-graphite/>

11 Biber et al. (2023) Improved production process with new spheroidization machine with high efficiency and low energy consumption for rounding natural graphite for Li-ion battery applications. Carbon, 201, 847-855.

12 Biber et al. (2023) Improved production process with new spheroidization machine with high efficiency and low energy consumption for rounding natural graphite for Li-ion battery applications. Carbon, 201, 847-855.

TECHNOLOGY STATE OF PLAY

Micronisation and spheronisation are mature processes currently used in the graphite supply chain, globally, and several emerging producers in Australia are piloting production.

- In February 2024, International Graphite commissioned a micronising plant at their downstream graphite processing facility in Collie, Western Australia.[13] Currently operating at pilot scale with a capacity of 200 tonnes per annum, the plant is set to become a commercial facility capable of handling 4,000 tonnes per annum.
- Australian-owned company Renascor Resources has collaborated with international companies, trialling their feedstock with commercial milling equipment. The trials will factor into the design of Renascor's planned spherical graphite manufacturing facility in South Australia, which will use graphite concentrates from their Siviour Project. In April 2024, the Government conditionally approved a $185 million loan from its Critical Minerals Facility to fast track the development this project.[14]
- Talga Technologies have a patented process for purifying graphite which includes a spheronisation stage.
- Finally, Australian based Ecograf have announced plans for a development facility in the Kwinana-Rockingham region of Western Australia, which includes micronisation and spheronisation equipment.

Research initiatives in Australia are aiming to overcome the key challenges of spheronisation. For example, the FBICRC's "Super Anode" has stated a goal to reduce spheronisation wastage of natural graphite by 30%.[15]

Beyond graphite, milling and classification is widely used across several other industries. Australia has a large METS sector and active research programs covering beneficiation, including milling and classification technologies. Cross-cutting capabilities can be leveraged and applied to the graphite space. For example, the University of Newcastle's Centre for Critical Minerals and Urban Mining focuses on developing technologies related to particles across several industries.[16] This includes patenting and commercialising the REFLUX® Classifier across several countries.[17]

An analysis of global patent activity from 2007 to 2022 identified that micronisation and spheronisation accounted for 4.4% of the patent families relating to graphite processing. There has been a consistent rise in activity with 30% of patent activity throughout the period. China (44%), Japan (26%) and the Republic of Korea (18%) were the major players in this space. Key Australian organisations with patents involving a micronisation and spheronisation step includes Talga resources.

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[13] International Graphite (2024) Collie Micronising Qualification Plant Commissioned. <https://www.internationalgraphite.com.au/collie-micronising-qualification-plant-successfully-commissioned/>

[14] Prime Minister of Australia (2024) Critical minerals funding helps deliver future made in Australia. Press Release. <https://www.pm.gov.au/media/critical-minerals-funding-helps-deliver-future-made-australia>

[15] Future Battery Industries CRC (n.d.) Super Anode. <https://fbicrc.com.au/project/super-anode/>

[16] The University of Newcastle (n.d.) Centre for Critical Minerals and Urban Mining. <https://www.newcastle.edu.au/research/centre/critical-minerals-urban-mining>

[17] FLSmidth (n.d.) REFLUX Classifier. <https://www.flsmidth.com/en-gb/products/centrifugation-and-classification/reflux-classifier>

The following table summarizes the key RD&D areas of focus in the micronisation and spheronisation of graphite:

Table 2: Global RD&D focus areas for the micronisation and spheronisation of graphite.

RD&D FOCUS AREAS	
Micronisation and spheronisation	• Improving the yield of the milling process, which is often as low as 35–50%.[18] • Developing graphite byproducts and exploring various value-added applications, including applications in polymer nanocomposites, conductive coatings, and graphene production. • Reprocessing of finer particles into particles within battery specifications.[19] • Improving processing time and electricity consumption.

3.1.2 Implications for Australia

Micronisation and spheronisation are essential processes used to produce spherical graphite particles from natural graphite, a key material for lithium-ion battery anodes. Well understood across the minerals industry, these processes represent a key value-adding opportunity for Australian graphite companies.

This section discusses the opportunities for domestic RD&D and international engagement in micronisation and spheronisation (summarised in Figure 8). More details on the framework used can be found in the main report *From minerals to materials: Assessment of Australia's critical mineral mid-stream processing capabilities.*

Figure 8: Opportunities for Australian RD&D and international collaboration in graphite processing.

Opportunity area	Establish new capability in emerging technologies	Accelerate emerging technologies and grow Australian IP	Pilot and scale up Australian IP	Support commercial deployment of mature technologies
Applicable Technologies				• Micronisation and spheronisaton
RD&D actions	Build capability in emerging technology areas via fundamental and applied research projects.	Leverage Australia's strengths to progress technologies beyond the lab and grow Australian IP.	Deploy Australian IP in pilot-scale and commercial-scale demonstrations.	Support the deployment of mature technologies domestically at commercial scale, through commercial testing and validation, and cross-cutting RD&D.
International engagement actions	Engage with research institutions on capability building and knowledge sharing (e.g. joint research programs).	Partner with overseas industry, research or government on mutually beneficial sustained technology development efforts (e.g. co-funded or joint projects).	Engage with upstream offtakers to de-risk and finance pilot projects. Alternatively, demonstrate Australian technologies overseas.	Engage on commercial arrangements e.g. international technology providers, license overseas patents, attract foreign direct investment, and secure offtake agreements.

Micronisation and spheronisation are highly mature but have faced a steady rise in global patent activity indicating there is some room for innovation. Enhancing the yield of current processes, a persistent

[18] Fischer S et al. (2023) Impact of Spheroidization of Natural Graphite on Fast-Charging Capability of Anodes for LIB. Batteries. <https://www.mdpi.com/2313-0105/9/6/305>

[19] Stakeholder consultations

challenge for producers, may have prompted the additional research which evident in the increased patent activity. Furthermore, developing methods that generate graphite particles fulfilling specific properties, including particle size, surface area and tap density is another area requiring innovation. Variation in these parameters influences the design and efficacy of the resulting anode and is a key demand of battery manufacturers. Lastly, there are challenges in scaling the process for higher production, typically resulting in an increase in the number of mills rather than their size.

Australia's activity in the space is predominantly commercial pilots of micronisation and spheronisation technologies on Australian flake graphite to support industrial scale-up. Due to the maturity of the technology and the accessibility of off-the-shelf equipment, domestic producers largely opt for utilising equipment developed by overseas manufacturers for pilot projects, rather than investing resources in developing their own intellectual property (IP) in this domain.

Australia has the opportunity to continue to engage in partnerships with foreign original equipment manufacturers (OEMs) to grow a domestic vertically integrated industry. Further, in the absence of a domestic battery manufacturer, collaboration with offtakers will be essential to align the specifications (e.g., size and shape) of the spherical graphite with the needs of end customers, ensuring a successful match in the market. To enable industry competitiveness there is a role for Australian RD&D to optimise the operation, subject to the ore characteristics and commercial equipment, with the goal of improving yields and efficiency, whilst ensuring production fulfils offtakers specifications.

3.2 Production of purified spherical graphite

After the initial processing steps, the micronised and spheronised graphite needs to achieve a purity level of approximately 99.95%. This stringent purity requirement is essential to guarantee high capacity and prolonged cycle life in LIBs. Impurities exceeding this threshold can trigger irreversible reactions that can adversely affecting cell performance and lifespan.[20] Graphite purification predominantly relies on two broad routes: hydrometallurgical and pyrometallurgical methods.

3.2.1 Hydrofluoric acid method

Chemical purification with hydrofluoric acid (HF) is a widely used technique used to achieve battery-grade graphite.[21] HF is known for its effectiveness in dissolving most impurities and organic materials, particularly silicate minerals commonly found in graphite deposits.[22] During the leaching process, HF generates water-soluble compounds, which can be removed through a water scrubbing process and subsequently dried.[23] However, the HF method requires significant control measures, of both the process and its effluents, due to the acid's corrosivity and toxicity.[24]

[20] Glass et al. (2023) Natural Graphite Purification Report. Future Battery Industries CRC.

[21] Zhao et al. (2022) High efficiency purification of natural flake graphite by flotation combined with alkali-melting acid leaching: application in energy storage. Journal of Materials Research and Technology 21, 4212–4223.

[22] Chelgani et al. (2016) A Review of Graphite Beneficiation Techniques. Mineral Processing and Extractive Metallurgy Review 37(1), 58–68; Yang et al. (2022) Purification mechanism of microcrystalline graphite and lithium storage properties of purified graphite. Materials Research Express 9(2), 025505; Syarifuddin et al. (2016) Effect of acid leaching on upgrading the graphite concentrate from West Kalimantan (Indonesia). In AIP Conference Proceedings (Vol. 1712, No. 1). AIP Publishing.

[23] Huang et al. (2022) Assessment of Spherical Graphite for Lithium-Ion Batteries: Techniques, China's Status, Production Market, and Recommended Policies for Sustainable Development. Advanced Sustainable Systems 6(11), 2200243.

[24] Al-Sairafi et al. (2022) Study on purification of flake graphite by heat activation and hydrofluoric acid. Advances in Materials and Processing Technologies 8(4), 4564–4578.

TECHNOLOGY STATE OF PLAY

Currently, the dominant method for graphite purification globally is the hydrofluoric acid method, particularly in China. The use of hydrofluoric acid is highly restricted in other countries and therefore graphite purification using this method is not widely conducted outside of China.

3.2.2 Hydrofluoric acid-free methods

Several alternatives to hydrofluoric acid have been developed to circumvent health, safety and environmental issues of hydrofluoric acid use. These include including high temperature methods, leaching using alternative acids, chlorination and alkali roasting.

High temperature methods

In high temperature methods, graphite ore is subjected to extreme temperatures of 2400 °C to 3000 °C in a furnace or in a fluidized bed reactor, vaporising impurities.[25] The heating can be undertaken in an inert atmosphere, such as nitrogen, to prevent the degrading of the mechanical and thermal properties of the graphite.

RD&D efforts in pyrometallurgical methods involve exploring techniques that to achieve high temperatures while optimising power efficiency and cost. Furthermore, batch processing used can be time and energy consuming (e.g., waiting for furnace to heat and cool).

Alternative acids

Due to the significant sustainability and health concerns of the HF leaching process, some RD&D has explored alternatives acids and/or a mixed acid approach. Acids used in these methods include sulfuric, hydrochloric and hexafluorosilicic acids.[26] A further benefit of the mixed acid approach is the ability to remove naturally grown impurities in graphite crystal gaps, such as pyrites.[27] However, challenges such as long reaction time, substantial acid consumption and pollution concerns persist.

Alkali-acid method

The alkali-acid approach for graphite purification involves subjecting graphite to a sequence of reactions using various bases and acids to form compounds that can be easily removed by washing with water. The process begins by reacting the graphite with sodium hydroxide (NaOH) at elevated temperatures. Here, the impurities react with the NaOH, leading to the formation of water-soluble hydroxides.[28] After this initial leaching process, the graphite is washed, returning it to neutral.[29] Following this, several different acids

[25] Jara et al. (2019). Purification, application and current market trend of natural graphite: A review. International Journal of Mining Science and Technology, 29(5), 671-689; Adham K, Bowes G (2018) Natural graphite purification through chlorination in fluidized bed reactor. Extraction 2018: Proceedings of the First Global Conference on Extractive Metallurgy; Lähde et al. (2024) Effect of high temperature thermal treatment on the electrochemical performance of natural flake graphite. Journal of Materials Research, 1-11.

[26] Oh J (2022) Purification of Graphite Concentrate by Leaching with Phosphorus Chemistry. McGill University, Canada; Yang et al. (2022) Purification mechanism of microcrystalline graphite and lithium storage properties of purified graphite. Materials Research Express, 9(2), 025505.

[27] Oh J (2022) Purification of Graphite Concentrate by Leaching with Phosphorus Chemistry. McGill University.

[28] Jara et al. (2019) Purification, application and current market trend of natural graphite: A review. International Journal of Mining Science and Technology 29(5), 671-689.

[29] Wang et al. (2017) Insights into alkali-acid leaching of sericite: dissolution behavior and mechanism. Minerals, 7(10), 196.

including hydrochloric acid or sulfuric acid can be used (depending on the impurities in the graphite) to form soluble metals species that are subsequently removed by water leaching.[30]

The alkali-acid leaching method is a complex process, with its success contingent on three key parameters: the alkali leaching temperature; sodium hydroxide concentration; and leaching time.[31] Furthermore, it encounters hurdles such as equipment corrosion, environmental pollution, and substantial energy consumption. Nevertheless, it offers advantages including low production costs, heightened efficiency, and high product quality.[32]

Chlorination roasting

Chlorine roasting can be operated at lower levels than high temperature methods, whilst still maintaining high purification and significant recovery rates.[33] This process involves heating graphite and subsequently introducing chlorine gas to remove the impurities in the graphite. Chlorine purification has to date been used mostly on synthetic graphite in a batch process.[34]

Challenges of chlorination roasting include equipment costs and managing toxic emissions and corrosivity. Fluidized bed reactors are being explored to enable continuous instead of batch processing, as well as support control over reagent consumption and process duration.[35]

Alkali roasting

An efficient method for purifying graphite involves alkali roasting. This process entails roasting the graphite alongside sodium hydroxide (NaOH), subsequently washing it with water, and then leaching it with an acid.[36]

This approach offers the advantage of achieving effective purification at relatively low roasting temperatures, typically between 200-900°C.[37] In addition to successfully removing silicate impurities, this process has been found effective at eliminating sulphide impurities as well.

However, a portion of graphite can be lost during alkali roasting, unless conducted in an inert environment.[38] Despite not attaining the same ultra-high purities as other high-temperature methods, alkali roasting meets the requisite specifications for battery-grade graphite materials.

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[30] Chelgani et al. (2016) A Review of Graphite Beneficiation Techniques. Mineral Processing and Extractive Metallurgy Review, 37:1, 58-68

[31] Wang et al. (2017) Insights into alkali-acid leaching of sericite: dissolution behavior and mechanism. Minerals, 7(10), 196.

[32] Guo et al. (2021) Research on Purification Technology of Ultra-Large Flake Graphite Based on Alkali-Acid Method. In Materials Science Forum, 1036, 104-113.

[33] Jara et al. (2019). Purification, application and current market trend of natural graphite: A review. International Journal of Mining Science and Technology 29(5), 671-689; Adham K, Bowes G (2018) Natural graphite purification through chlorination in fluidized bed reactor. Extraction 2018: Proceedings of the First Global Conference on Extractive Metallurgy.

[34] Adham K, Bowes G (2018) Natural graphite purification through chlorination in fluidized bed reactor. Extraction 2018: Proceedings of the First Global Conference on Extractive Metallurgy.

[35] Adham K, Bowes G (2018) Natural graphite purification through chlorination in fluidized bed reactor. Extraction 2018: Proceedings of the First Global Conference on Extractive Metallurgy.

[36] Luv J, Forssberg E (2002) Preparation of high-purity and low-sulphur graphite from Woxna fine graphite concentrate by alkali roasting. Minerals engineering 15(10), 755-757.

[37] Oh J (2022) Purification of Graphite Concentrate by Leaching with Phosphorus Chemistry. McGill University.

[38] Wang et al. (2018) A novel technique for microcrystalline graphite beneficiation based on alkali-acid leaching process. Separation Science and Technology 53(6), 982–989.

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Due to the considerable regulation of large-scale hydrofluoric acid use in most countries (including Australia),[39] many companies are testing the viability of alternative pyrometallurgical and hydrometallurgical purification methods.[40] The high temperature method is also mature and used at commercial scales globally.[41]

Pathways utilising alkaline reagents (alkali roasting and alkali-acid methods) are being developed and applied at lab scales globally, with several Australian companies currently considering scaling them up.

- Renascor Resources Ltd. has conducted tests on their graphite using a three-step purification technique involving caustic roasting and sulphuric acid leaching. The process is able to achieve a 99.99% purity level.[42]
- Mineral Commodities has conducted lab-scale testing on two purification processes - a chlorination process (carbochlorination) and an alkaline process (caustic roasting – hydrochloric acid leaching) – for their proposed Active Anode Material Plant in Norway. These processes both achieved a battery-grade purity level of 99.95%.[43]
- Ecograf, with graphite deposits in Tanzania, has developed a multi-stage caustic baking and leaching purification method that can achieve a purity level of 99.95% and above.[44]

Outside of Australia, Sarytogan Graphite conducted purification test work for its sample from Central Kazakhstan in collaboration with Pro-Graphite (Germany). The test work achieved a purity level of 99.70% after alkali roasting, and 99.87% when combining alkali roasting with sulphuric acid leaching. The company is assessing the potential of alternative acids to increase the purity level.[45]

Graphite purification through chlorination roasting remains at pilot scale.

- As mentioned previously, Mineral Commodities has conducted lab-scale tests for chlorination roasting.
- Outside of Australia, Nouveau Monde Graphite (Canada) has reported producing graphite at 99.95% purity at its demonstration plant in Quebec using a proprietary carbochlorination process.[46]

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[39] Work Health and Safety Regulations (2011) – Schedule 15. Australian Government. <https://www.legislation.gov.au/F2011L02664/latest/text>; Safe Work Australia (2024) Major hazard facilities. <https://www.safeworkaustralia.gov.au/safety-topic/industry-and-business/major-hazard-facilities>

[40] Loh J (2022) Powering Australia: how CSIRO is helping industry meet demand for graphite. CSIRO. <https://www.csiro.au/en/work-with-us/industries/mining-resources/resourceful-magazine/issue-27/graphite-processing-for-batteries>

[41] Chen Q (2022) High Temperature Continuous Gas Purification and Graphitization System and Process for Carbon Particulate Materials. In Journal of Physics: Conference Series 2152, 1. <https://news.metal.com/newscontent/102602431/>

[42] Renascor Resources (2023) Siviour Battery Anode Material Study Results. <https://renascor.com.au/wp-content/uploads/2023/08/20230808-Siviour-Battery-Anode-Material-Study-Results-2588185.pdf>

[43] Mineral Commodities (2020) MRC Completes Pre-Feasibility Study for Active Anode Material Plant in Norway, Addressing the Fast Growing Battery Market. <https://www.mineralcommodities.com/wp-content/uploads/2020/09/PFS-for-Active-Anode-Plant-in-Norway.pdf>

[44] EcoGraf (2023) Corporate Presentation. <https://www.ecograf.com.au/wp-content/uploads/2023/07/2576821.pdf>; AU2021261902 B2 (EcoGraf) (2020) Method of producing purified graphite.

[45] Sarytogan Graphite (2022) Breakthrough: 99.87% graphite purity. <https://announcements.asx.com.au/asxpdf/20221206/pdf/45jgy96wrl1v4c.pdf>

[46] Allaire et al. (2022) NI 43-101 Technical Feasibility Study Report for the Matawinie Mine and the Bécancour Battery Material Plant Integrated Graphite Projects. Nouveau Monde Graphite, p. 48 – 49; Nouveau Monde Graphite (2021) Nouveau Monde Provides Corporate Update on the Development of its Mine-to-Market Battery Material Business. Press release. <https://nmg.com/corporate-update-q3-2021/>.

Finally, Magnis Energy Technologies (Australia) has adopted a mechanical process (consisting of flotation) without any requirements for chemical or high temperature purification, due to the nature of their unique graphite deposit in Tanzania.[47]

A global patent analysis of the 2007 to 2022 period identified 4.9% of patent families were related to purification processes. There has been a steep increase in activity from 2019 onwards, reflecting the interest in for high-purity graphite for battery applications. Within the analysed period, China (85.7%) and the United States (8.9%) were the major players in this space. Australian companies that have produced patents in purification methods include Ecograf and CSIRO, both in alkaline processes.

The following table summarizes the key RD&D areas of focus in the purification of graphite:

Table 3: Global RD&D focus areas for the purification of graphite.

RD&D FOCUS AREAS	
Alternative acids	• Reducing reaction times and acid consumption and managing pollution (e.g. use of less aggressive or recyclable reagents).
Alkali-acid method	• Reducing complexity and processing time, lowering costs. • Optimise energy efficiency, streamline processes, and reduce water consumption. • Managing the toxic wastes and emissions associated with chemical use.
High temperature methods	• RD&D efforts are focused on reducing electricity consumption and costs associated with very high temperature processes, as well as the low production scale currently possible.
Chlorination roasting	• Developing and scaling application of chlorination to natural graphite, reducing reagent consumption, accelerating reaction times, enabling continuous processing.[48] • Developing mitigation and control measures for corrosivity and expelled gases.[49]
Alkali roasting	• Utilising alternative acids (e.g., hydrochloric acid) and recycling the waste liquid to reduce the environmental impact of the process.[50] • Overcoming graphite oxidation loss by further lowering the temperatures required while reacting with sodium hydroxide (e.g., 150 – 270°C).[51]

[47] Magnis Energy Technologies (n.d.) Anode active materials. <https://magnis.com.au/anode-active-material/>.

[4848] Adham K, Bowes G (2018) Natural graphite purification through chlorination in fluidized bed reactor. Extraction 2018: Proceedings of the First Global Conference on Extractive Metallurgy.

[49] Adham K, Bowes G (2018) Natural graphite purification through chlorination in fluidized bed reactor. Extraction 2018: Proceedings of the First Global Conference on Extractive Metallurgy.

[50] Ri H, Ri K, Kim K, Ri K, Yu J, Pak K, Choe D, Kang S and Hong S (2022) Effective purification of graphite via low pulp density flotation-low temperature alkali roasting-acid leaching route: From laboratory-scale to pilot-scale. Minerals Engineering 188, 107852.

[51] Wang et al. (2018) A novel technique for microcrystalline graphite beneficiation based on alkali-acid leaching process. Separation Science and Technology 53(6), 982–989.

3.2.3 Implications for Australia

High-purity graphite is essential for LiB battery applications, emphasising the significance of purification for anode material production. Given global supply chain concentration, there is an opportunity for Australia to undertake purification onshore. However, current practices have significant environmental challenges and more sustainable processes will be needed to enable safe domestic production.

This section discusses the opportunities for domestic RD&D and international engagement in graphite purification (summarised in Figure 9). More details on the framework used can be found in the main report *From minerals to materials: Assessment of Australia's critical mineral mid-stream processing capabilities*.

Figure 9: Opportunities for Australian RD&D and international collaboration in graphite purification.

Opportunity area	Establish new capability in emerging technologies	Accelerate emerging technologies and grow Australian IP	Pilot and scale up Australian IP	Support commercial deployment of mature technologies
Applicable Technologies			• Alkali roasting • Alkali-acid method • Chlorination roasting • High temperature method	• High temperature method
RD&D actions	Build capability in emerging technology areas via fundamental and applied research projects.	Leverage Australia's strengths to progress technologies beyond the lab and grow Australian IP.	Deploy Australian IP in pilot-scale and commercial-scale demonstrations.	Support the deployment of mature technologies domestically at commercial scale, through commercial testing and validation, and cross-cutting RD&D.
International engagement actions	Engage with research institutions on capability building and knowledge sharing (e.g. joint research programs).	Partner with overseas industry, research or government on mutually beneficial sustained technology development efforts (e.g. co-funded or joint projects).	Engage with upstream offtakers to de-risk and finance pilot projects. Alternatively, demonstrate Australian technologies overseas.	Engage on commercial arrangements e.g. international technology providers, license overseas patents, attract foreign direct investment, and secure offtake agreements.

IP, intellectual property.

Despite its commercial maturity, graphite purification is an active area of R&D internationally, indicating opportunities for innovation. The inherent shortcomings of the mature hydro/pyrometallurgical purification methods have likely sparked renewed interest in purification, which was evident in the patent analysis from 2019 onwards. These include the use of environmentally hazardous substances such as hydrofluoric acid, the energy demands required to reach temperatures of up to 2700 °C, as well as challenges in meeting the specified purity standards for battery-grade graphite. This underscores the importance of developing innovative purification techniques that achieve a purity level surpassing 99.9% carbon in cost and environmentally friendly ways.

Ultimately, diversifying graphite purification outside of China will require the development hydrofluoric (HF) acid-free methods due to its high risks, and Australia is well positioned to do so with strong RD&D and patent output. Australia has developed patents across several alternatives to HF-acid, such as chlorination roasting and alkaline roasting or leaching pathways. Continued scale up of alternative purification pathways (that are HF-acid-free) is required beyond the laboratory and pilot scales. Further technical improvements, testing and economic feasibility studies can ensure the graphite can achieve the required purity at scale, whilst remaining profitable.

In order to enable diversified commercial production, Australian purification technologies should be developed for both domestic operations and for graphite deposits internationally (some of which are held by Australian companies). Offtake agreements and enduring relationships with key battery manufacturers overseas will be essential to domestic graphite producers. It is important to acknowledge the rigorous qualification processes that battery manufacturers require to ensure the product meets the specific quality and safety standards. Australian C-SPG companies entering the market must work closely with battery and/or automotive manufacturers and establish enduring relationships in order to be considered for procurement. International co-operation with countries and companies with high sustainability standards and goals to incorporate eco-friendly standards into the graphite supply chain, will be important. This can facilitate Australia's position as a strategic and sustainable supplier.

3.3 Production of coated purified spherical graphite (C-SPG)

Carbon coating is an integral step in the graphite supply chain, aimed at improving the performance of natural graphite anode materials, and therefore the performance of lithium batteries.[52] The carbon layer prevents the formation of an excessive SEI (solid electrolyte interface) during battery cycling, buffers the expansion of the material, and improves conductivity. [53] Publicly known methods to produce coated spherical graphite include wet chemical processing and dry processes, usually followed by high temperature thermal treatment. [54] Various carbon sources - including pitch, tar, bitumen, asphalt and resin – can be used as a coating material in this process. These viscous materials are derived from petroleum or coal by-products and have a high carbon content.[55] Various additives and dopants are often applied to coatings to further enhance performance. However, this is a highly proprietary area.

3.3.1 Wet processes

In wet chemical processes, the carbon source is applied to the graphite particles with the help of a solvent (e.g., a tetrahydrofuran, hexane, or toluene solution).[56] The purified graphite and the carbon source are added to a suitable liquid medium in which they can be dispersed and thoroughly mixed through stirring, centrifugation (spinning) or using ultrasonic frequencies (sound waves). The mixture is dried to evaporate the liquid medium and is subjected to multiple heat treatments (500 °C to 1,000°C) to consolidate the carbon coating. The resulting carbon-coated graphite is then pulverised and screened to obtain particles of the required size.[57]

Challenges include implementing alternative solvents that remain effective at dispersing the coating agent while being cost-effective and less hazardous; and controlling the characteristics of the coating layer to optimise anode performance. [58]

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[52] Kwon et al. (2021) Achieving High-Performance Spherical Natural Graphite Anode through a Modified Carbon Coating for Lithium-Ion Batteries. Energies, 14(7), 1946; Kim B, Kim J, Im J (2022) Effect and Mechanism of Pitch Coating on the Rate Performance Improvement of Lithium-Ion Batteries. Materials 15(13), 4713.

[53] Yaroslavtsev B, Stenina A (2020) Carbon coating of electrode materials for lithium-ion batteries. Surface Innovations 9(2–3), 92-110.

[54] Han et al. (2015) Coating of graphite anode with coal tar pitch as an effective precursor for enhancing the rate performance in Li-ion batteries: Effects of composition and softening points of coal tar pitch. Carbon 94, 432-438.

[55] Kim B, Kim J, Im J (2022) Effect and Mechanism of Pitch Coating on the Rate Performance Improvement of Lithium-Ion Batteries. Materials 15(13), 4713.

[56] Jo YJ and Lee JD (2019) Effect of petroleum pitch coating on electrochemical performance of graphite as anode materials. Korean Journal of Chemical Engineering 36 (10), 1724–1731.

[57] Han et al. (2015) Coating of graphite anode with coal tar pitch as an effective precursor for enhancing the rate performance in Li-ion batteries: Effects of composition and softening points of coal tar pitch. Carbon 94, 432-438; Zhang et al (2018) Effective regeneration of anode material recycled from scrapped Li-ion batteries. Journal of Power Sources 390, 38–44.

[58] Liu et al. (2020). Rapid coating of asphalt to prepare carbon-encapsulated composites of nano-silicon and graphite for lithium battery anodes. Journal of Materials Science 55(10), 4382–4394; Kim et al. (2023) Optimization of the molecular weight range of coating pitch and its effect on graphite anodes for lithium-ion batteries. Korean Journal of Chemical Engineering 40(12), 2839–2846.

3.3.2 Dry processes

In dry processes, purified graphite and the carbon source are mixed while in their solid state, eliminating the need for solvents. Coating occurs when the dry particles are mixed under mechanical assistance (e.g. using a rotary mixer, hot press, jet mill, or centrifuge).[59] The solid mixture undergoes a heat treatment (approximately 900 °C) to form a carbon shell around the graphite particle, with pulverisation and screening used for particle size control depending on the carbon source and mixing process used.[60]

The main drawback of dry processes is the lower precision and control over uniformity and thickness.[61]

3.3.3 Alternative processes

Chemical vapour deposition (CVD) is another process that has been used to produce C-SPG.[62] This continuous process uses high temperatures and exposes the material to be coated to a hydrocarbon gas that serves as the carbon source. The gas reacts at the surface of the material, forming a thin film on top of it.[63] A variant of this process, atomic layer deposition (ALD), utilises multiple gaseous precursors to sequentially react with a surface, enabling precise control over coating thickness and uniformity with each gas pulse depositing a single layer.[64]

Despite enabling control and uniformity, CVD and ALD require a specialised setup (e.g., fluidised bed reactor) for use at scale, and the loss of unreacted gas reagents can make it inefficient.[65]

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Processes for coating purified spherical graphite are currently being used at commercial scale outside Australia.

- For example, the Graphex Group uses an asphalt coating to produce C-SPG (10,000 metric tonnes per annum) at its factory in Jixi City (China).[66]

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[59] Jo YJ, Lee JD (2019) Effect of petroleum pitch coating on electrochemical performance of graphite as anode materials. Korean Journal of Chemical Engineering 36 (10), 1724–1731; Kim et al. (2022) Effect and Mechanism of Pitch Coating on the Rate Performance Improvement of Lithium-Ion Batteries. Materials 15(13), 4713.

[60] Kim et al. (2022) Effect and Mechanism of Pitch Coating on the Rate Performance Improvement of Lithium-Ion Batteries. Materials 15(13), 4713; Liu et al. (2020) Rapid coating of asphalt to prepare carbon-encapsulated composites of nano-silicon and graphite for lithium battery anodes. Journal of Materials Science 55(10), 4382–4394.

[61] Sharova et al. (2017) Evaluation of Carbon-Coated Graphite as a Negative Electrode Material for Li-Ion Batteries, C 3(4), 22; Li H, Zhou H (2012) Enhancing the performances of Li-ion batteries by carbon-coating: present and future. Chemical Communications 48(9), 1201–1217.

[62] Ding et al. (2006) Characteristics of graphite anode modified by CVD carbon coating. Surface and Coatings Technology 200(9), 3041–3048

[63] Sun et al. (2021) Chemical vapour deposition. Nature Reviews Methods Primers 1(1):5; Oka et al. (2022) Effect of amorphous carbon coating on the formation of solid electrolyte interphase and electrochemical properties of a graphite electrode. Journal of Power Sources 543:231850.

[64] National Renewable Energy Laboratory (n.d.) Battery Materials Synthesis. Transportation & Mobility Research. <https://www.nrel.gov/transportation/battery-materials-synthesis.html>; Forge Nano (2022) So you think EV batteries are green? <https://www.forgenano.com/so-you-think-ev-batteries-are-green/>.

[65] Li H, Zhou H (2012) Enhancing the performances of Li-ion batteries by carbon-coating: present and future. Chemical Communications 48(9), 1201–1217; Wang et al. (2023) Hydrothermally Deposited Carbon Coatings for Li-Ion Battery Active Materials. Journal of The Electrochemical Society 170(8), 080518.

[66] Graphex Group (2023) Coated Spherical Graphite. Products. <https://graphexgroup.com/products/coated-spherical-graphite/>; Graphex Group Limited (2022) Amendment No. 7 To Form F-1 Registration Statement Under The Securities Act Of 1933 - Preliminary Prospectus. United States Securities and Exchange Commission. Washington, D.C. <https://www.sec.gov/Archives/edgar/data/1816723/000110465922084721/tm2118847-24_f1a.htm#tOUIN>

- Meanwhile, Imerys Graphite & Carbon employs CVD at its production site in Kitakyushu (Japan) to produce carbon-coated anode materials.[67]
- Emerging alternatives like ALD are also advancing towards commercial scale implementation. Pilot and small commercial scale systems are being developed by the US company Forge Nano and tested in collaboration with international graphite producers.[68]

Within Australia, projects have not focussed on C-SPG specifically, but have explored other types of coatings for graphite, demonstrating a general capability for coating methods. For example, in 2020 Altech Batteries Ltd collaborated with Curtin University and the University of Western Australia to demonstrate its technology for coating graphite with a layer of high-purity alumina (HPA).[69] The company has also focussed its Australian R&D on coating silicon particles using its HPA technology and is currently building a pilot plant to produce coated anode material in Saxony, Germany.[70]

Outside of graphite, Australia has demonstrated strong capabilities in coating technologies more generally, particularly thin films, as these have cross-cutting applications across multiple industries. For example, the Melbourne Centre for Nanofabrication (a part of the Australian National Fabrication Facility) is a joint venture between eight Australian research institutions supporting innovation in advanced materials. This includes thin film facilities (e.g., ALD and CVD).[71] Industry-research collaborations on thin films include the University of South Australia's partnership with SMR Automotive[72] and the biomedical spinout TekCyte.[73]

An analysis of global patent filing activity from 2007 to 2022 found 7.2% of patent families were related to the carbon coating of graphite. It is likely that this figure underestimates the level of activity in this domain, given the inherently proprietary nature of the field. However, since 2019, a noticeable upswing in activities was observed. The top countries by output include China with 54.9% of patent families, The Republic of Korea with 20.7% and Japan with 17.1%. Australian organisations with IP involving carbon coating of graphite material includes Talga.

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[67] Imerys (2023) Graphite. Minerals & more. <https://www.imerys.com/minerals/graphite>; Imerys (2022) Imerys Graphite & Carbon Company Profile. <https://www.imerys.com/public/2022-06/imerys-business-documentation-graphite-and-carbon-company-profile-june-2022.pdf>

[68] Forge Nano (2022) So you think EV batteries are green? <https://www.forgenano.com/so-you-think-ev-batteries-are-green/>; Nouveau Monde (2020) Nouveau Monde and Forge Nano Sign a Collaboration Agreement for Advanced Coatings for Li-Ion Battery Anode Material. News. <https://nmg.com/nouveau-monde-and-forge-nano-sign-a-collaboration-agreement-for-advanced-coatings-for-li-ion-battery-anode-material/>

[69] Altech Chemicals Limited (2020) Altech - Confirmation of HPA Graphite Particle Coating Technology. ASX Announcement and Media Release. <https://announcements.asx.com.au/asxpdf/20201222/pdf/44r6nk4hyljfgt.pdf>

[70] Altech Batteries Limited (2023) German Battery Materials Coating Plant. Projects - German Battery Materials Coating Plant. <https://www.altechgroup.com/projects/german-battery-materials-coating-plant/>

[71] Melbourne Centre for Nanofabrication (n.d.) Thin films. <https://nanomelbourne.com/services/thin-films/>

[72] University of South Australia We partnered with the auto industry to create the world's first shatterproof car mirrors. <https://www.unisa.edu.au/research/impact-stories/shatterproof-car-mirrors/>

[73] TekCyte (2023) Medical Device Coating. <https://tekcyte.com/medicaldevicecoating/>

The following table summarizes the key RD&D areas of focus in the production of coated spherical graphite:

Table 4: Global RD&D focus areas for the production of coated spherical graphite.

	RD&D FOCUS AREAS
Coated spherical graphite production	• Optimising the surface uniformity of coated particles, to increase anode durability and performance (e.g. improving control of pitch addition or advancing controllable, thin layer deposition methods).[74] • Identify and trial alternatives to petroleum and coal by-products that can serve as economically viable carbon sources for coating processes. • Develop and assess dopants embedded in the coating layer and the coating of composite materials, to explore potential improvements to anode performance.[75] • Develop the use of coating technologies to reinstate the properties and performance of spent graphite, as part of its recycling process.[76] • Trial and implement alternatives to hazardous and costly solvents used in some wet chemical processes.[77] This can include replacements that pose lower risk but remain suitable to disperse graphite and the carbon source.

3.3.4 Implications for Australia

Coating represents the final step in an integrated graphite material operation and is an opportunity for Australia to realise the full economic opportunities of domestic anode material production. Coated purified spherical graphite (C-SPG) attracts a significantly high price compared to uncoated material. Further, this is a highly concentrated part of the global supply chain, representing an opportunity for Australia to be an alternative supplier.

This section discusses the opportunities for domestic RD&D and international engagement in graphite coating (summarised in Figure 10). More details on the framework used can be found in the main report *From minerals to materials: Assessment of Australia's critical mineral mid-stream processing capabilities*.

[74] Kim B, Kim J, Im J (2022) Effect and Mechanism of Pitch Coating on the Rate Performance Improvement of Lithium-Ion Batteries. Materials 15(13), 4713.

[75] Liu W, Xu H, Qin H, Lv Y, Zhu G, Lei X, Lin F, Zhang Z, Wang L (2020) Rapid coating of asphalt to prepare carbon-encapsulated composites of nano-silicon and graphite for lithium battery anodes. Journal of Materials Science 55(10), 4382–4394.

[76] Zhang et al. (2018) Effective regeneration of anode material recycled from scrapped Li-ion batteries. Journal of Power Sources 390, 38–44; Ma Z, Zhuang Y, Deng Y, Song X, Zuo X, Xiao X, Nan J (2018) From spent graphite to amorphous sp 2 +sp 3 carbon-coated sp 2 graphite for high-performance lithium ion batteries. Journal of Power Sources 376, 91–99.

[77] Liu W, Xu H, Qin H, Lv Y, Zhu G, Lei X, Lin F, Zhang Z, Wang L (2020) Rapid coating of asphalt to prepare carbon-encapsulated composites of nano-silicon and graphite for lithium battery anodes. Journal of Materials Science 55(10), 4382–4394.

Figure 10: Opportunities for Australian RD&D and international collaboration in graphite coating.

Opportunity area	Establish new capability in emerging technologies	Accelerate emerging technologies and grow Australian IP	Pilot and scale up Australian IP	Support commercial deployment of mature technologies
Applicable Technologies				• Carbon coating
RD&D actions	Build capability in emerging technology areas via fundamental and applied research projects.	Leverage Australia's strengths to progress technologies beyond the lab and grow Australian IP.	Deploy Australian IP in pilot-scale and commercial-scale demonstrations.	Support the deployment of mature technologies domestically at commercial scale, through commercial testing and validation, and cross-cutting RD&D.
International engagement actions	Engage with research institutions on capability building and knowledge sharing (e.g. joint research programs).	Partner with overseas industry, research or government on mutually beneficial sustained technology development efforts (e.g. co-funded or joint projects).	Engage with upstream offtakers to de-risk and finance pilot projects. Alternatively, demonstrate Australian technologies overseas.	Engage on commercial arrangements e.g. international technology providers, license overseas patents, attract foreign direct investment, and secure offtake agreements.

Coating processes generally employ highly mature synthesis technologies; however, technology maturity appears to differ depending on jurisdiction. Coating technology and coating formulations are a highly proprietary area, and highly subject to trade secrets. This is a substantial barrier for countries and emerging graphite companies seeking to enter the market for C-SPG. Despite maturity, an upswing in patent activity since 2019 indicates this is an area with potential for innovation. The need to develop coating activity outside of concentrated locations is also a strong justification for technology development.

Despite Australia's lack of patents in the C-SPG space, Australia's research sector has shown strong activity in coatings and thin film technology generally for the production of advanced materials. Building domestic capability in carbon coating for C-SPG anodes will require leveraging Australia's skills in advanced materials, coupled with collaboration with global industry partners, such as international battery cell manufacturers or automotive companies, and manufacturing equipment suppliers. Strong co-operation will be required with international battery manufacturers, as coating formulas are often highly proprietary and subject to trade-secrets and need to meet battery manufacturer specifications.

3.4 Production of silicon-graphite compound materials

Composite materials consisting of silicon and graphite are increasingly being used to enhance the performance of lithium-ion batteries combining the high capacity of silicon and the conductivity of carbon. Although silicon significantly increases the energy density of batteries, its tendency to undergo substantial volumetric expansion causes the solid electrolyte interface (SEI) to become unstable, degrading the performance of the battery.[78] Manufacturers have been delivering breakthroughs in the amounts of silicon incorporated, pushing the boundaries of battery performance. Silicon-graphite composites, where the silicon and graphite sit within a matrix structure, offer a solution to buffer the expansion of the silicon.[79] This section will explore the several methods of synthesising these materials.

[78] Chen X, Yang W, Zhang Y (2021) Advanced Electrode Materials for Lithium-ion Battery: Silicon-based Anodes and Co-less-Ni-rich Cathodes. In Journal of Physics: Conference Series 2133. IOP Publishing.

[79] Mueller et al. (2021) Si-on-Graphite fabricated by fluidized bed process for high-capacity anodes of Li-ion batteries. Chemical Engineering Journal 407, 126603.

3.4.1 Mechanical ball milling

Mechanical ball milling is a mature, low-cost technology which can be used to synthesise silicon-graphite composite materials for anodes. This is achieved by mixing silicon and graphite powders (amongst other additives, such as aluminium and nickel) in a ball mill. Mechanical pressure uniformly fragments and distributes the silicon and graphite to form a composite material. [80]

This method benefits from being low-cost, [81] and easily electrified however there is less control over particle uniformity and properties compared to other methods. Additional steps, such as thermal or chemical treatment, can be employed to optimise the composite properties,[82] however this adds to energy and chemical costs.

3.4.2 Wet chemical processes

Wet chemical processing is another route used to synthesise silicon-graphite composite materials, whereby the silicon, graphite and chosen additives are mixed into a solution.[83] The solution is subsequently evaporated off at low temperatures, prior to further heat treatment in a furnace.[84]

Wet chemical processes are being investigated due to their simplicity, making them easy to scale. [85]

3.4.3 Spray-based methods

Spray drying is an alternative method for preparing silicon-graphite composite anode materials. In this process, a slurry or a solution containing graphite and silicon (and other additives) is sprayed and heated to high temperatures to form a composite powder.[86] Spray drying processes are relatively low-cost and have high throughput and scalability.[87] Furthermore, the processes provide better control over the spherical morphology and size of the composite materials. [88]

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3.4.4 Chemical vapor deposition

Chemical vapor deposition (CVD) is a widely used technique in several industries where a solid material is vaporised and deposited onto the surface of another material.[89] The technique can be used to make silicon-graphite composites which can be customised by adjusting parameters like the precursor, reaction rate, and temperature used.[90] Using this method, porous silicon can be coated with a layer of carbon. Alternatively, a silicon nanolayer can be deposited onto graphite.[91]

CVD techniques are being developed for a silicon-graphite composite anode materials due to their ability to offer a high degree of control and versatility for coatings.[92]

TECHNOLOGY STATE OF PLAY

Silicon-doped graphite anodes are becoming more commonplace in the electric vehicles market. In 2022 composite anodes (comprising 5–10% silicon content) accounted for approximately 30% market share, showing steady increases in silicon content in the past five years.[93] Commercial examples include the Panasonic 2170 cell which incorporates roughly 5% silicon and has been used in various Tesla models since 2015.[94]

Automotive manufacturers are actively pursuing higher silicon ratios to maximise energy and cost improvements.[95] In fact, some companies have cited the goal of completely removing graphite from their batteries. Anodes with silicon content greater than 10% are at lower levels of technology maturity for EV LIBs, ranging from lab to pilot scale.

- For example, OneD Battery Sciences (US) has produced silicon-graphite composite materials with up to 25% silicon content at pilot scale and has a dedicated facility in Washington state.[96]

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- Also in the US, Sila Nanotechnologies is producing a nanocomposite anode material that reportedly comprises 50% silicon at its pilot facility in Alameda, California.[97] The company is building a commercial scale plant to provide its material to EV manufacturers.[98]
- Finally, Amprius (US) is already commercialising LIBs with 100% silicon anodes for use in specialty applications, particularly aerospace.[99] Unlike other approaches that rely on spherical nanoparticles, Amprius' anode is composed by porous silicon nanowires that can withstand swelling.[100]

In Australia, AnteoTech and Sicona Battery Technologies Pty Ltd are developing silicon-graphite composite anode materials that offer advantages in storage capacity and cost effectiveness, as well as improving the weight and size of batteries.[101] Sicona prepare their composite material via a spray drying method, followed by heat treatment.[102]

- Sicona's silicon-graphite composite is currently at pilot scale, with production based at the company headquarters and pilot facility in Wollongong.[103] The company is working towards establishing commercial production in the US, with plans for a 5kt per annum plant.[104]
- AnteoTech's silicon composite anode is currently at lab scale.[105] Limited information is available on AnteoTech's synthesis method.
- Finally, Talga Group's silicon-graphene-graphite anode has progressed from lab scale tests in UK to a pilot plant in Germany.

Silicon-graphite composite materials attracted significantly more attention than other areas covered in the report, making up 83.5% of patents in graphite processing from 2007 - 2022. Global patent output has experience strong growth over time peaking between 2019 and 2020, reflecting technology trends observed in the lithium battery market. The top countries by output include China with 60.3% of patent families, the Republic of Korea with 18.1% and Japan with 7.5%. Australian organisations with patent activity in silicon-graphite composites include the University of Wollongong, Talga, Sicona and Anteo.

The following table summarizes the key RD&D areas of focus for the production of silicon-graphite compounds:

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Table 5: Global RD&D focus areas for the production of silicon-graphite compounds.

RD&D FOCUS AREAS	
General	• Development of material structures and compositions that overcome the limits to silicon content (relating to silicon's expansion during cycling), and that can be effectively fabricated at scale.
Mechanical methods	• Improving morphology control and uniformity across the composite material.
Wet chemical methods	• Expanding the range of material structures obtained, and other material improvements (e.g. using dopants or additional carbon structures like nanotubes), to increase anode stability and performance. [106]
Spray-based methods	• Improving the thermal efficiency of the process at scale (e.g., heat recovery strategies, or increasing product yield).[107]
Chemical vapour deposition	• Reducing the energy intensity of this process and managing the environmental or safety risks associated with some of the gases that may be used in this method (e.g., silane in the case of silicon deposition).[108]

3.4.5 Implications for Australia

Developing scalable fabrication processes to produce Si/C composite anodes that surpass lithium battery performance limits and reduce costs is a significant economic opportunity for technology developers, prompting significant RD&D efforts in the industry and research sectors. Given the economic opportunity for this technology, momentum in this domain is strong. This is reflected in the strong growth of patent output in silicon-graphite composites, and their volume relative to other areas of graphite processing.

This section discusses the opportunities for domestic RD&D and international engagement in silicon-graphite compound materials (summarised in Figure 11). More details on the framework used can be found in the main report *From minerals to materials: Assessment of Australia's critical mineral mid-stream processing capabilities*.

[106] Li et al. (2021) Diverting exploration of silicon anode into practical way: a review focused on silicon-graphite composite for lithium ion batteries. Energy Storage Materials 35, 550-576

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[108] Pedersen et al. (2021) Green CVD—Toward a sustainable philosophy for thin film deposition by chemical vapor deposition. Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films 39(5); Liu et al. (2021) Large-Scale Production of a Silicon Nanowire/Graphite Composites Anode via the CVD Method for High-Performance Lithium-Ion Batteries. Energy & Fuels 35(3), 2758–2765.

Figure 11: Opportunities for Australian RD&D and international collaboration in silicon graphite compound materials.

Opportunity area	Establish new capability in emerging technologies	Accelerate emerging technologies and grow Australian IP	Pilot and scale up Australian IP	Support commercial deployment of mature technologies
Applicable Technologies		• Silicon-graphite compound materials	• Silicon-graphite compound materials	
RD&D actions	Build capability in emerging technology areas via fundamental and applied research projects.	Leverage Australia's strengths to progress technologies beyond the lab and grow Australian IP.	Deploy Australian IP in pilot-scale and commercial-scale demonstrations.	Support the deployment of mature technologies domestically at commercial scale, through commercial testing and validation, and cross-cutting RD&D.
International engagement actions	Engage with research institutions on capability building and knowledge sharing (e.g. joint research programs).	Partner with overseas industry, research or government on mutually beneficial sustained technology development efforts (e.g. co-funded or joint projects).	Engage with upstream offtakers to de-risk and finance pilot projects. Alternatively, demonstrate Australian technologies overseas.	Engage on commercial arrangements e.g. international technology providers, license overseas patents, attract foreign direct investment, and secure offtake agreements.

IP, intellectual property.

Advancements in silicon-graphite composites are essential to ensure that anode materials achieve a prolonged cycle life for LIBs, whilst addressing issues such as volume expansion and ensuring a high-level of safety in consumer applications. Central to this is overcoming challenges associated with solid electrolyte interphase (SEI), which becomes damaged with swelling that occurs in the charging and discharging of silicon anodes. One area of innovation involves engineering the particle structure, to minimise or remove the effects of silicon swelling on the electrolyte.[109] Common RD&D themes across the different composites production methods include improving morphology control and uniformity, as well as enhancing the versatility of the materials.

Australia has strong RD&D activity in this emerging area, including multiple Australian based organisations that have patented silicon-graphite anode technologies. With Australia's strength in this area, there is scope to leverage existing capabilities in silicon-graphite anode materials, working towards the commercialisation of next generation technologies. Piloting and demonstrating Australian technologies should not be done in isolation, given Australia does not currently have any large battery or automotive manufacturers. Establishing international partnerships will be important for Australian technology to enter the global EV market.

[109] Sila (2020) The future of energy storage: Towards a perfect battery with global scale. White paper. <https://www.silanano.com/uploads/Sila-_-The-Future-of-Energy-Storage-White-Paper.pdf>

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