

Journal Review: Developments in TiSi₂ Material Synthesis and Application Potential in Defence Industry Sector

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Abstract

Titanium disilicide (TiSi₂) is a promising intermetallic material due to its unique properties, such as high temperature oxidation and corrosion resistance, good thermal and electrical conductivity, and low density. In this review article, various aspects of TiSi₂ are discussed, including the properties and characteristics of the material, synthesis methods, applications, and development opportunities. The method used in writing this article is the Systematic Literature Review (SLR) by searching, collecting, and evaluating several sources related to the research object. Recent development in TiSi₂ applications include semiconductors (Salicide technology which has lower resistivity), longer-lasting lithium-ion battery anodes, high temperature resistant coatings and components, hydrogen generators and defence system components. With the potential to be produced locally using Indonesian resources, such as Ilmenite and silica sand, TiSi₂ can promote self-sufficiency of the national strategic industry, including in the defence and energy sectors.

Keywords: Domestic Industry, Metallurgical Waste, Semiconductor, TiSi₂



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INTRODUCTION

TiSi₂ is intermetallic silicide compound formed of refractory metal and silicon. TiSi₂ has two common phases: metastable C49 with Body-centered Orthorhombic structure, and stable C54 with Face-centered Orthorhombic structure. C54 has better resistivity and can be sintered at higher temperature than C49 (Liao, 2006). Titanium is a refractory metal, a group of metals that are very resistant to heat and wear. The 5 elements known as the main refractory metals, with melting point above 2000°C, are Molybdenum, Niobium, Tantalum, Tungsten, and Rhenium. Sometimes, the classification is expanded to include 9 other elements with melting point above 1850°C: Vanadium, Chromium, Zirconium, Hafnium, Ruthenium, Rhodium, Osmium, Iridium, dan Titanium. Refractory metal is used for high temperature applications such as jet turbine and nuclear reactor (Snead et al., 2019).

Silicon part of silicide has important roles, especially in high temperature because of oxidation process. When oxidized, silicide such as TiSi₂ formed protective SiO₂ coating, hindering consequent oxygen diffusion. SiO₂ is highly desired protective coating for its adherent nature and high melting point reaching 1710°C (Strydom et al., 1985). Oxide layer formed on top of TiSi₂ is protective and adherent, even after undergoing exposure to high and fluctuating temperatures for long periods (known as cyclic oxidation), with little sign of damage (Chaia et al., 2015). These properties make TiSi₂ an attractive material for applications at high temperature conditions.

However, one challenge of silicide materials application is that although these materials are strong at high temperatures (>1000°C), they have limitations at moderate temperatures (400-800°C); At this temperature, there is possibility of pesting oxidation; a process where the material disintegrates due to the large increase in volume of the material oxide, creating

internal stress on the structure. This is the biggest weakness of MoSi₂, silicide commonly used for high strength, high temperature applications. Its oxide, MoO₃, has a large volume and a melting point of only 795°C (Volders & Reinke, 2019). TiSi₂, on the other hand, forms layers of more stable TiO₂ and SiO₂ (with more SiO₂ formed at temperatures above 1300°C,) (Becker et al., 1992). TiSi₂, as an intermetallic material, can be combined with other materials with different phases to obtain the desired properties due to the material phase and microstructure formed. MoSi₂, other silicide of refractory metal, and different silicide of titanium like Ti₅Si₃, are possible materials to be combined with TiSi₂. Mixed nanocomposites of TiSi₂ and MoSi₂ can be modified to obtain the desired properties if combined with materials such as aluminium, because within the crystal lattice, aluminium can replace the silicon atoms in MoSi₂ and titanium on TiSi₂. The presence of aluminium will facilitate the formation and filling of vacancies in the microstructure (Všianská et al., 2023). Theoretically, this makes it easier to research and engineer new alloys using MoSi₂/TiSi₂ with certain desired properties.

RESEARCH METHODS

This research uses comprehensive literature study approach, using sources from scientific journals, books and relevant news. Journal sources are taken from reputable peer-reviewed journals to maintain validity. Some recent developments on the synthesis and applications of TiSi₂ explained and compared with existing synthesis methods and applications. These developments are then linked to the situations and opportunities in Indonesia by citing recent news.

RESULTS OF RESEARCH AND DISCUSSION

Development of TiSi₂ synthesis

Table 1. Various synthesis process of TiSi₂ and the application sectors

Process	Resulting TiSi ₂	Application	References
Self-propagating high-temperature synthesis (SHS)	Powder or bulk	material and metallurgy engineering	Xu et al., 2021
Rapid Thermal Annealing (RTA)	Contact material for semiconductor	Semiconductor	Kim et al., 2023
Field Emission	nanowire	Semiconductor	Xiang et al., 2005
Shock loading	bulk	photocatalyst	Liu et al., 2013
Sintering	bulk	material and metallurgy engineering	Sinurat, et al, 2024

TiSi₂ can be synthesized in various ways, usually by mechanically activating titanium and silicon powder through mechanical milling process until evenly mixed. The resulting powder is then processed into TiSi₂ compound, and further processed according to needs. Examples of TiSi₂ synthesis processes is described in table 1. Self-propagating high-temperature synthesis (SHS) heats the powder, and the energy from the exothermic process will spread to other molecules until all the materials combine to form TiSi₂ (Xu et al., 2021). Rapid thermal annealing (RTA) reacts titanium thin films with silicon substrates at temperatures of 700-1000°C (Kim et al., 2023). Field emission create nanowire on the surface of silicon substrate by depositing titanium powder then heated in tubular furnace (Xiang et al., 2005). Shock loading introduce sudden mechanical force on powder created through mechanical milling, creating bulk material. The resulting bulk material is excellent photocatalyst semiconductor; in breaking down water molecules into hydrogen and oxygen gas, photocatalyst with material that have been through both mechanical milling and shock loading, perform better than material that have been through only mechanical milling or shock loading. The study conclude that both processes mechanically activate the material, and combining both produce an even mixture and

a smoother microstructure, thereby increasing the surface area for reaction (Liu et al., 2013). While sintering is a process that apply heat and pressure to the powder material until it solidifies without reaching the melting point. The heat is to activate the materials to be tightly packed together. The author is currently researching TiSi₂ synthesized with Pulsed Electric Current Sintering process, where the heat is provided through pulsed DC Electric current through a conductor to produce a denser solid material. The schematic of the process and the resulting sample is shown in figure 1.

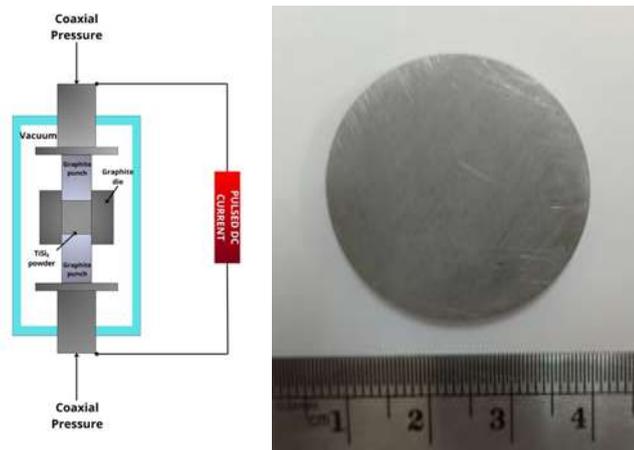


Figure 1. Sintering schematic and the TiSi₂ sample created

Note in these methods, TiSi₂ is synthesized from raw titanium and silicon materials with a relatively high level of purity. However, titanium and silicon can be found in other forms and compositions. One of the breakthroughs in the synthesis of TiSi₂ is by processing Ti-Bearing Blast Furnace Slag (TBFS). TBFS is a metallurgical industrial waste obtained after processing iron sand, and because of its complex structure and low grade, become too complicated to be processed (Y. Zhang et al., 2023). Currently, TBFS produced by the metallurgical industry in China has reached millions of tons and causes environmental pollution (Y. Zhang et al., 2022), while the titanium contained, up to almost 20% of the TBFS mass, is left unutilized. Y. Lei, Y. Zhang et al. is one of the research groups that has attempted to innovate TBFS processing to extract value out of this waste. This process is carried out by adding low purity silicon (99.3%) to TBFS, the mixture is then heated at a temperature of 1500°C in a 99% argon atmosphere, to separate the Ti-Si alloy from TBFS (Lei et al., 2019). The Ti-Si alloy can then be separated using a directional magnet into a Si/TiSi₂ alloy and high purity Si solid (99.7%) (Li et al., 2021, 2022). Not only TiSi₂, the high purity Si obtained from this process will also be useful in various sectors, giving this new process high economic value (Y. Zhang et al., 2022).

TiSi₂ application as Salicide for semiconductors

Table 2. Comparison of CoSi₂ dan TiSi₂ Salicide properties

Properties	CoSi ₂	TiSi ₂
Resistivity	10-25 μΩ/cm	12-24 μΩ/cm
Metal-dopant compound formation	No	Yes
Thermal expansion	10 ppm/°C	12 ppm/°C
Mechanical stress	(8-10) x 10 ⁹ dyne/cm ²	(2-2.25) x 10 ¹⁰ dyne/cm ²
Si consumption (nm Si/nm Silicide)	1.03	0.904
Reaction temperature with SiO ₂	> 1000 °C	700 °C
Deposition	Difficult	Easy
Sheet resistance control	Good	Poor
Resistivity to dry/wet etching	Good	Poor

Native oxide consumption	Poor	Good
Lattice match with Si	Good	Bad
Melting point	1325 °C	1540 °C

Table 3. Lithium-ion battery anodes comparison

Delithiation phase	Lithiation phase	Structure	Volume change	Potential vs. Li/Li+
Graphite	1/6 LiC ₆	Layered	10%	0.3 V (← → LiC ₆)
Li ₄ Ti ₅ O ₁₂	Li ₇ Ti ₅ O ₁₂	Spinel	1%	0.87 V
Si	Li _{3.75} Si	Amorphous	263%	0.2 V (→ Li ₁₂ Si ₇)
β-Sn	Li _{3.75} Sn	Crystal	190%	0.3 V (← → Li ₁₃ Sn ₅)
Al	Li ₉ Al	Crystal	180%	0.12 V (← → LiAl)

Silicide materials have unique properties that make them useful as micro electric semiconductors Self-Aligned Silicide or abbreviated salicide. This technology is created by reacting silicon with a thin film of metal in the active region of a semiconductor. Because it is self-aligned, this semiconductor will have smaller resistivity as the process prevents the formation of parasitic resistivity (resistance that does not exist in the design, and arises due to the micro electric manufacturing process) (Ekström & Zetterling, 2023). TiSi₂ is one of the materials that's often used for salicide due to the low resistivity of TiSi₂ C54. TiSi₂ C49 can be converted into TiSi₂ C54 through Rapid Thermal Annealing process in temperature 625-700°C (Mann et al., 1993). Developments of TiSi₂ application as salicide is currently driven by the need for mass production of small-sized semiconductors along with the development of micro electric technology. Current process of mass production of salicide creates unwanted resistivity at the contact area between the thin film and the silicon. Table 2 shows a comparison of the properties between the two commonly used salicide, CoSi₂ and TiSi₂. The comparison shows that salicide TiSi₂ is easier to synthesize, but CoSi₂ tend to have better performance (Liao, 2006; Wei et al., 1989). Mass production of TiSi₂ salicide would be easier but with limited performance. Kim M. et al. found, by adding a layer of tantalum (Ta) in the silicidation process, there's an increase in diffusion barrier properties, including reduced electrical resistance and improved thermal stability. These characteristics result in TiSi₂ salicide to be more reliable in extreme conditions, such as on supercomputer devices. (Kim et al., 2023). This innovation will also reduce some of the electrical weaknesses of the TiSi₂ that have been mentioned, turning TiSi₂ into promising contact material for the future.

TiSi₂ as an anode material for Lithium-ion batteries

TiSi₂, that has good conductivity, also have applications outside semiconductors, such as battery electrodes. Lithium ion (Li-ion) batteries are a type of rechargeable battery that is widely used in electronic devices, electric vehicles, and energy storage systems. These batteries work by transferring lithium ions between the anode (can be made of silicon) and cathode (usually made of lithium compounds) during charging and use. Silicon is an interesting material to use as an anode material because it has a higher capacitance than carbon anodes (such as the widely used graphite). The challenge is the in a process called lithiation, where the anode material reacts with lithium ions during energy charging, and delithiation, when lithium ions are released again during use. Silicon weakness compared to other type of anodes is highlighted in Table 3; upon charging, lithium and silicon ions form an amorphous Si-Li alloy, accompanied with a large volume change (263% of the initial size) (Mishra et al., 2024), dislocations will occur and in the long term, damage the silicon anode's microstructure. Then when the energy is used, the lithium ions will move and the size of the silicon compound will shrink again, but plastic deformation can occur in the anode which reduces the battery life. To reduce the

occurrence of this process, silicon material is combined with other materials to form composites, such as TiSi_2 .

Currently, nano-sized composite materials are being developed from silicon and TiSi_2 (Si/TiSi_2) to be applied to lithium-ion batteries. L. Wang et al. create composites using molten salt electrolysis process, which has a hollow structure to reduce deformation and atomic dislocation because there is space during lithiation. Si/TiSi_2 has proven to be effective as an anode material because of conductivity and mechanical resistance, especially at higher temperatures, so that when used, the plastic deformation from lithiation-delithiation that occurs can be reduced or prevented (Wang et al., 2024). Y. Zhang focuses on the economical angle, namely synthesizing this composite material by recycling TBFS with the low purity silicon mentioned earlier in this article. It was found, increasing TBFS when synthesizing Si/TiSi_2 composites initially will limit the initial capacity of the battery due to the additional material/impurities content from TBFS. However, after several cycles of use, the composite made from more TBFS will have the smallest capacity reduction due to the presence of TiSi_2 (Y. Zhang et al., 2021). Xu et al. forgoes using silicon as a base but instead created TiSi_2 anodes wrapped in Reduced Graphene Oxide (RGO) to increase conductivity. TiSi_2 is synthesized with "chemical oven" self-propagating high-temperature synthesis (COSHS); where a mixture of titanium and silicon powder, mixed using mechanical milling process, is heated with "chemical oven" in the form of a mixture of Ti & C as a heat propagation medium. After cleaning contaminants, obtained TiSi_2 has high purity and no other titanium silicides such as Ti_5Si_3 or TiSi were detected. When used as an anode, TiSi_2 shows good energy storage capacity due to the lithiation process, TiSi_2 reacts with lithium ions to form $\text{TiSi} + \text{SiLi}_2$, which exceeds TiSi_2 's normal capacity. This provides an opportunity for silicide materials to become an alternative to commonly used graphite electrodes (Xu et al., 2021).

TiSi_2 as a material with high temperature resistance

In designing composite or ceramic materials for aeronautical, space, nuclear and energy applications, components of various shapes and thicknesses are needed. To simplify and cheapen manufacturing and production process, components with complex shapes are composed of smaller components that are glued together. The adhesive material must also have high temperature resistance. Spurred by the Columbia space shuttle disaster in February 2003, extensive worldwide research was conducted to develop a high-temperature resistant ceramic adhesive, which was used to repair cracks and holes in the space shuttle's wings and nose during manned missions. adhesives make a major contribution to structural integrity, ease of manufacture, improved performance and safety, as well as cost and time savings. TiSi_2 , is one of the active ingredients types of filler that has been proven to increase the strength and quality of adhesive joints (Mahajan & Johnson, 2020). TiSi_2 is one of the lightest silicide materials ($4,02 \text{ g/cm}^3$) but is still stronger than other light silicides, such as VSi_2 ($4,42 \text{ g/cm}^3$) and CrSi_2 ($4,91 \text{ g/cm}^3$). Although all three have poor oxidation resistance at moderate temperatures, among the three materials TiSi_2 shows the best oxidation resistance at temperatures of 450°C to 950°C , and has the lowest mass increase due to oxidation even after long-term exposure of up to 1000 hours (Chaia et al., 2015). Resistance at moderate temperatures is very important because components are not constantly exposed to high temperatures. Apart from joints and adhesives, the application of composite materials containing TiSi_2 include use case as a high temperature resistant coating material, especially because of its lightweightness and oxidation resistance. TiSi_2 have high temperature oxidation resistance due to the formation of SiO_2 , which adheres tightly to the material surface, withstands high temperatures, and does not consume a lot of material. The result is a component that is highly durable because little wear is caused by

oxidation and a strong protective coating that prevents spalling due to exposure to heat and the environment (J. He et al., 2020).

TiSi₂ as a hydrogen generator component

Not only as a defence material, TiSi₂ There are also developments in the area of energy security. C. Zhang et al. developing MoS₂/TiSi₂ materials through an in situ photoreduction process, to produce photocatalytic (splitting water into hydrogen and oxygen using light) hydrogen (H₂) under simulated sunlight. TiSi₂ enhances photocatalytic activity by increasing the rate of charge separation. MoS₂/TiSi₂ composite shows significant potential for efficient and sustainable H₂ production, and has adequate durability (C. Zhang et al., 2019).

TiSi₂ as raw or composite material for defence and security instruments

TiSi₂ can also be combined with other materials because of its good mechanical properties even under extreme conditions. D. Kozi n et al. in their research explored ceramic matrix composites produced through reactive sintering of boron carbide with Ti-Si intermetallic compounds, such as TiSi₂. This material exhibits high hardness, thermal stability, and oxidation resistance, making it suitable for defence applications such as armor systems and protective coatings. TiSi₂ contributes to increased toughness and structural integrity in extreme conditions, critical requirements for mission and defence performance, with better hardness than additions Ti₅Si₃ (Kozi n et al., 2022). Carbon fibre reinforced carbon (C_f/C) is a composite material that is light, durable and has resistance to high temperatures, so it is attractive for application in various situations, with the main obstacle being how to combine this material with other materials without reducing its performance, for example in heavy vehicle hinges which receive heat radiation from machine. Addition of TiSi₂ in the carbon fiber composite will produce a hinge that has the toughness and crack resistance of TiSi₂, tensile and compressive strength of carbon fibre, as well as resistance to high temperatures such as oxidation and corrosion, while remaining light and durable (Z. He et al., 2020).

Potential Sources and Applications in Indonesia

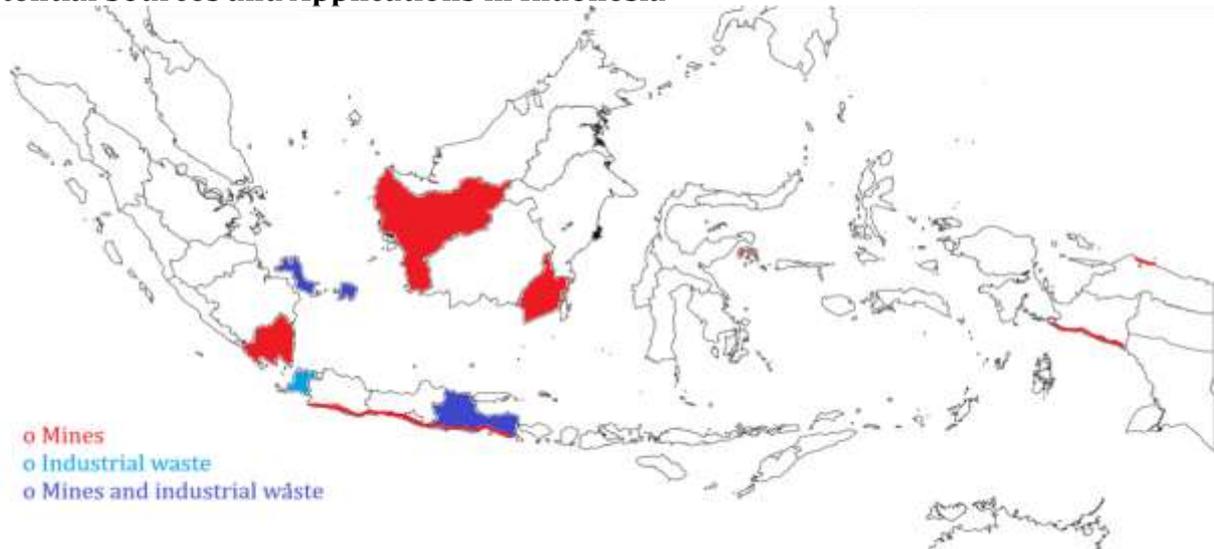


Figure 2. Titanium sources in Indonesia can come from iron sand mines and industrial waste

Breakthrough in synthesis of TiSi₂ materials will have an impact on the self-sustainability of the domestic industries supply chain (including the defence industry) through raw materials downstreaming, with locally produced TiSi₂ can be used as local content for defence vehicles.

Low purity silicon can be processed from silica sand which is abundant in Indonesia (Sasongko & Agung Dwi E, 2024), and TBFS from the processing of iron sand, which contains Ilmenite (FeTiO_3). Seen in Figure 3, the locations of Ilmenite reserves in Indonesia are found in West Kalimantan, South Kalimantan, the South Coast of Java from West Java to East Java, Bangka Belitung, and the Banggai Islands, Central Sulawesi (Geosriwijaya.com, 2024). TBFS itself is also found as waste of iron sand processing from the metallurgical industry, which is found in Cilegon, Banten, and Lumajang, East Java. (Pratiwi, 2023). Seeing opportunities from downstream resources including titanium processing, the government has also started construction of the first titanium smelter in Indonesia, located in the Bangka Belitung, even if it was hampered by Covid-19. (Maranda, 2023). Once operational, the titanium smelter will process ilmenite into titanium that can be further processed into raw materials needed for the production of medical devices, military equipment and aircraft raw materials (Yogatama, 2023). Bahfie et al. found that the iron sand content in Indonesia was detected to contain enough ilmenite to be magnetically separated quite easily (Bahfie et al., 2022). The separation results can then be reduced selectively, such as using the method with TBFS and silicon discussed above.

The development of semiconductors will encourage the Indonesian semiconductor and electronics industry to be more independent, and can be used to make equipment that can be used reliably even in less-than-ideal conditions, such as on the battlefield, where there is far from maintenance. Currently, Indonesia is still limited to the mining of raw materials, and the assembly, testing and packaging of semiconductors. Coordinating Minister for Economic Affairs Airlangga Hartanto said Indonesia will develop the semiconductor industry, with the United States and Japan to become co-investor for semiconductor industry in Indonesia (Simanjuntak, 2024). Batteries are also currently a trending technology, especially due to the scarcity of some materials that only exist in certain countries, such as more than 80 percent of the world's lithium being mined in Australia, China and Chile. China also controls more than half of the world's lithium processing and 75 percent of lithium-ion battery manufacturing (Balkus, 2022). This is significant because one of these materials is nickel, controlled by Indonesia. Thus, independence of battery industry is considered something of great concern, for example with the government announce a plan to become the top 3 electric vehicle battery producers in the world by 2027 (Medina, 2024). A breakthrough in making lithium-ion batteries that are more resistant to lithiation and de-lithiation will strengthen Indonesian battery industry, by increasing the quality, not only the quantity, of batteries produced. Improved qualities include durability, that also useful on military deployments where supply chains are difficult to reach. Hydrogen generator innovations could support alternative energy uses for military vehicles, drones and portable energy storage. Efficient H_2 production process can facilitate decentralized energy generation, especially in remote areas or conflict areas where fuel supplies will be difficult, as well as a sustainable energy source to reduce dependence on imported energy sources. TiSi_2 is also a candidate for advanced/cutting-edge materials. Composite material containing TiSi_2 can be applied in extreme conditions such as high temperature, acid and corrosiveness environment and has high durability, allowing it to increase the quality and service life of national defence and security equipment.

CONCLUSION

In this article TiSi_2 and its new discoveries are discussed, especially in the last 5 years. The discoveries being talked about ranges from synthesize process of the material and their applications, such as in semiconductor sectors, energy storage and generation, and high temperature conditions. Highlighted are the potential applications of these breakthroughs in

Indonesia, especially in the defence sector, both for military platforms/vehicles and the potential for defence industry, and supply chain independence. By producing TiSi₂ locally, Indonesia has the potential to reduce dependence on imports for important defence technology, including turbine engines for fighter jets. Thus, TiSi₂ can contribute to strategic industrial growth while supporting sustainability in material development.

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