Recycling of the Diamond-wire Saw Powder by Ni-catalyzed Nitridation to Prepare $\text{Si}_3\text{N}_4$

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Abstract
In this paper, the acceleration effect of nickel (Ni) on the direct nitridation process of the diamond-wire saw powder (DWSP) was investigated. The DWSP doped with Ni additives were nitrided at different temperatures. To study the mechanism of accelerated nitridation, the thermodynamics of Si-O-N-Ni and nitridation processes were analyzed by FactSage 7.2 and single-crystal silicon blocks were also nitrided instead of the DWSP. The results revealed that Ni decreased the nitridation temperature at which the DWSP began to gain significant weight and exhibited an excellent accelerating effect on the nitridation of the DWSP. At 1300°C the DWSP containing 2.0 wt% Ni additives had been completely nitrided within 2 h, whereas the DWSP without Ni additives had not been nitrided yet. Based on the equivalent substitution experiment, it could be concluded that the presence of Ni additives accelerated the nitridation and promoted the formation of the $\alpha$-$\text{Si}_3\text{N}_4$ nanorods through facilitating the generation of the SiO(g) and destructing the silica film on the surface of silicon at lower temperature. Meanwhile, Ni additives also played an important part in the growth of $\alpha$-$\text{Si}_3\text{N}_4$ nanorods by forming liquid Ni-Si alloy in the product.

Keywords Diamond-wire saw powder · Nickel catalyst · Nitridation · Catalytic mechanism · Silicon nitride

1 Introduction
In recent years, the photovoltaic industry has grown rapidly. However, in the process of producing silicon wafers for solar cells, the diameter of diamond wire is close to the thickness of the silicon wafer, resulting in nearly 40 % of the diamond-wire saw powder being produced and thrown away [1–4]. The DWSP mainly contains Si and SiO$_2$, with a small amount of metal impurities, such as Ca, Al, Fe, Ni and so on [5, 6]. It has extremely fine particle size and is a PM2.5 pollutant, which can cause safety and environmental problems. Therefore, corresponding treatment is extremely required to recycle it.

To solve the problems above mentioned, a lot of efforts on the recycle of the DWSP had been carried out, among which recycling high purity silicon had achieved remarkable results [7–11]. In addition, there are many feasible applications of the DWSP, in which the preparation of silicon nitride powder used as releasing agent or reusable nitride crucibles for crystalline silicon casting has quite potential [6, 12]. Silicon nitride is also a promising inorganic non-metallic material because of its excellent physical and chemical properties, such as high-temperature strength, thermal shock resistance, oxidation resistance, abrasion resistance, and self-lubrication [13, 14]. However, the direct nitridation of the DWSP has the disadvantages of higher nitridation temperature and longer nitridation time, which will lead to a huge waste of energy [15]. Therefore, some researches on the rapid nitridation of the DWSP had been carried out, one of which was to accelerate the nitridation of the DWSP by using metal additives [16–18]. Nevertheless, by the reason of researches on the nitridation of the DWSP by the metal additives promoted is quite small, the mechanism by which metal additives promote the nitridation of the DWSP is still unclear.

Thus, in this study, the acceleration of Ni in the direct nitridation process of the DWSP was studied detailedly. Besides, the corresponding mechanism of accelerated nitridation was also deduced properly based on the results.
This work provides a scientific basis for the preparation of silicon nitride at low temperature by the nitridation of diamond-wire saw powder.

2 Materials and Methods

2.1 Raw Material

The DWSP (Henan Xin Da New Material Co., LTD, China) and nickel powders (99.9 % pure, ≤ 100nm, Shanghai Aladdin Bio-Chem Technology Co., LTD, China) were used as the main experimental materials. Single-crystal silicon blocks (99.999 % pure, Hebei Xin Tie Metal Material Co., LTD, China) were used for the equivalent substitution experiment. High purity N₂ (99.999 % pure, Institute of Metal Research, Shenyang, China) was used as the gas source.

2.2 Processing Procedures

First, in order to avoid the influence of other metals, all metal impurities in the DWSP must be removed by acid pickling. Then, the DWSP after pickling and nickel powders were uniformly mixed for 8 h in a certain proportion (0.5, 1.0, 1.5, 2.0 wt% Ni) by the wet mixing ball milling process. Next, the mixed solution was dried at 65°C in the blast oven for 20 h to get rid of the organic solvent. Last, the DWSP doped with various content Ni were loosely placed into the crucible (45 × 22mm) and then put it into the constant temperature zone of a tube furnace. Before heating, purifying the tube furnace with nitrogen flow for 30 min at 80 ml/min to remove the air in the furnace. Then, the tube furnace was heated up to experimental temperatures at a rate of 5°C/min. After nitridation, keeping nitrogen flowing until the samples cooled with the furnace to the normal temperature. Nitridation experiments on single-crystal silicon blocks after surface polishing and oxidation treatment were consistent with the DWSP.

2.3 Characterization and Measurement

Thermogravimetric analysis was carried out to measure the weight changes in the nitrogen atmosphere. The thermodynamics of Si-O-N-Ni and nitridation processes were analyzed by FactSage 7.2. X-ray diffraction (XRD; D8 Advance, Bruker, Germany) was used to detected the crystalline phases compositions of the samples. Field-emission scanning electron microscopy (FE-SEM; Quanta250FEG, FEI Co., Ltd. Czechia), along with energy dispersive spectrometry (EDS; Oxford Instrument, UK) was used to observed the microstructures and morphologies of the samples. The Rietveld refinement method was adopted to calculated the relative content of each crystalline phase.

3 Results and Discussion

3.1 Thermogravimetric Analysis

The nitridation behavior of the DWSP before and after doping with Ni was studied through thermogravimetric analysis under a nitrogen atmosphere, and the test temperature rose from 25 to 1460°C with a heating rate of 10°C/min, as shown in Fig. 1. It could be found that the variation trend of both were similar, but the mass gain of the DWSP doped with Ni was significantly higher than that of the direct nitridation of the DWSP. In addition, both TG curves had obvious inflection points, and the mass began to increase significantly when the test temperature exceeded the inflection point temperature. By comparison, the temperature corresponding to the inflection point of the mass gain curve of the DWSP doped with Ni was 1325°C, which was lower than the temperature (1408°C) corresponding to the inflection point of the TG curve without Ni. These results indicate that Ni decreased the temperature at which the DWSP began to gain weight significantly.

3.2 Nitridation of the DWSP without Ni Additives

Figure 2a shows the XRD patterns of the DWSP after nitridation at different temperatures for 2 h. At 1300°C, it was clear that only Si (JCPDS No. 99 – 0092) peak was detected, indicating the DWSP had not been nitrided at this temperature evidently. On increasing the temperature to 1350°C, Si₃N₄ (α-Si₃N₄: JCPDS No.70-3756; β-Si₃N₄: JCPDS No. 71–0623) and tiny Si₂N₂O (JCPDS No. 84-1813) peaks were detected, but unreacted Si peaks maintained a high intensity, indicating the nitridation extent of the DWSP was still very low. With increasing the temperature to 1400°C, the intensity of Si₃N₄ peaks increased evidently, which indicates that the DWSP without Ni additives could effectively form Si₃N₄ only when the temperature reached 1400°C. Upon

Fig. 1 TG curves of the DWSP with and without Ni additives
further increasing the temperature to 1450°C, Si peak disappeared, indicating the DWSP could be completely nitrided at 1450°C for 2 h. When the temperature was improved to 1500°C, the phase compositions of the sample had no obvious changes.

Corresponding relative content of the phase compositions of the samples were calculated and shown in Fig. 2b. There was no Si₃N₄ formed at 1300°C, and only 56.1 wt% of Si₃N₄ was formed at 1350°C. When improving the temperature from 1350°C to 1400°C, the content of Si₃N₄ increased to 80.5 wt% whereas the content of unreacted Si decreased to 9.2 wt%. When the nitridation temperature reached to 1450°C, residual Si in the nitridation product disappeared, indicating only when the nitridation temperature reached at least 1450°C, the DWSP without additives could be completely nitrided. Finally, when the temperature was increased to 1500°C, there were no obvious changes.

3.3 Nitridation of the DWSP with Different Contents of Ni Additives

Figure 3a illustrates the XRD patterns of the DWSP doped with various contents Ni additives after nitridation at 1300°C for 2 h. Obviously, only Si peak was detected in the samples without Ni additives. When the sample only containing 0.5 wt% Ni, the phases composition changed evidently, Si₃N₄ and a small amount of Si₂N₂O were identified. With increasing the Ni additives to 1.5 wt%, Si peak decreased until it disappeared gradually, indicating the DWSP had been completely nitrided. With the further increase of Ni content to 2.0 wt%, the sample phases had no obvious changes.

Corresponding relative content of the phase compositions of the samples were shown in Fig. 3b. When the content of Ni additives increased from 0 wt% to 0.5 wt%, unreacted Si decreased from 100 wt% to 12.4 wt% at 1300°C. Meanwhile, α-Si₃N₄, β-Si₃N₄ and Si₂N₂O increased from 0 wt% to 63.3 wt%, 10.2 wt% and 14.1 wt%, respectively. On increasing Ni content to ≥ 1.5 wt%, unreacted Si in the sample decreased to 0 wt%. These results indicate the significant accelerating effect of Ni on the DWSP nitridation. Obviously, Si₂N₂O was detected in the samples whether it doped with Ni additives or not, there were two reasons: one was the high content of SiO₂ in the DWSP (about 17 wt%) [23] and the other was the trace oxygen contained in high-purity nitrogen.

Figure 4 presents typical SEM images of the samples doped with 0–2.0 wt% Ni after nitridation at 1300°C for 2 h and the corresponding EDS results. As shown in Fig. 4a and b, the morphology of the product without Ni did not change significantly and kept the slice-like structure of the DWSP. Only Si and O elements were detected in the product by EDS (Fig. 4c), indicating the DWSP had not been nitrided evidently, which was coincide with the previous XRD results shown in Fig. 3a. When the DWSP doped with 1.0 wt% Ni, the morphology of the product changed significantly, as shown in Fig. 4d and e. Many nanorods appeared in the product. Short nanorods with a diameter of about 25 nm were distributed on and around the surface of the granular products, and long nanorods with a diameter of about 60 nm were interspersed between the products. With increasing the Ni content from 1.0 wt% to 2.0 wt%, the quantity and of nanorods increased and most of the nanorods also increased to 60-200nm in diameter. It had been confirmed that [19, 20] these granular products were Si₃N₄ formed by gas-solid reaction and these nanorods were α-Si₃N₄ formed by gas phase reaction. This indicates that a large amount of gas was produced during nitridation. However, O elements had also been detected in the granular product and nanorods except Si and N elements.
Fig. 3 XRD patterns (a) and relative contents of the sample phases (b) of the DWSP doped with different contents of Ni additives after nitridation at 1300°C for 2 h.

Fig. 4 SEM images of the DWSP doped with various amounts of Ni: (a, b) 0wt%, (d, e) 1.0wt% and (g, h) 2.0 wt%, after nitridation at 1300°C for 2 h, and corresponding EDS results (c, f and i) of point A, B and C.
by EDS (Fig. 4f and i). It could be attributed to the surface of Si$_3$N$_4$ was oxidized into Si$_2$N$_2$O and the presence of SiO$_2$ amorphous layer [21, 22].

3.4 Nitridation of the DWSP with 2.0 wt% Ni Additives at Different Temperature

Figure 5a illustrates the XRD patterns of the DWSP doped with 2.0 wt% Ni additives after nitridation at different temperatures for 2 h. At 1200°C, unreacted Si peaks still maintained a high intensity, and weak Si$_3$N$_4$ and Si$_2$N$_2$O peaks appeared. On increasing the temperature to 1250°C, the intensity of Si$_3$N$_4$ and Si$_2$N$_2$O gradually increased, while Si peaks became significantly weaker. On further increasing the temperature to 1300°C, Si peaks disappeared, indicating the DWSP doped with 2.0 wt% Ni additives was completely nitrided at this temperature. With increasing the temperature from 1300°C to 1500°C, Si$_2$N$_2$O peak decreased whereas other peaks did not change evidently.

Corresponding relative content of the phase compositions of the samples were shown in Fig. 5b. At 1200°C, the content of unreacted Si reached 63.6 wt%, indicating only a small portion of the DWSP was nitrided. On increasing the temperature to 1250°C, unreacted Si in the sample decreased to 3.3 wt% whereas the content of Si$_3$N$_4$ increased from 27.1 wt% to 72.2 wt%. With increasing the temperature to 1300°C, there was no unreacted Si remained in the sample, indicating the complete nitridation of the DWSP. In contrast, the DWSP without Ni additives had not been nitrided yet at 1300°C, which indicates that Ni significantly promoted the nitridation of the DWSP and reduced the nitridation temperature. With the temperature further increased from 1300°C to 1500°C, the mass fraction of Si$_2$N$_2$O decreased from 19.6 wt% to 5.9 wt%, it could be attributed to the decomposition of Si$_2$N$_2$O [23].

3.5 Analysis of Ni Accelerated Nitridation Mechanism

The predominance area diagrams of Si-O-N-Ni system at 1523 K and 1623 K were conducted with FactSage 7.2, as shown in Fig. 6. In this work, the pressure in the furnace was 1.0 × 10$^{-1}$ MPa and the purity of nitrogen was 99.999%, so the nitrogen and oxygen pressure in the furnace was approximately 1 × 10$^{-1}$ MPa and 1 × 10$^{-6}$ MPa, respectively. The partial pressure of oxygen was much higher than that of the stable presence of Si$_3$N$_4$ and Si$_2$N$_2$O in the equilibrium state, so there would be no Si$_3$N$_4$ and Si$_2$N$_2$O formed, which was inconsistent with the previous nitridation results. Therefore, the trace oxygen in nitrogen participated in some reaction and was consumed.

Thermodynamic analysis proved that under the condition of P(N$_2$) of 1 × 10$^{-1}$ MPa, different oxygen partial pressures correspond to different products. As the partial pressure of oxygen increased, Si$_3$N$_4$ was gradually oxidized into Si$_2$N$_2$O and eventually into SiO$_2$. And it could be found that in the stable regions of Si$_2$N$_2$O and Si$_3$N$_4$, Ni$_x$Si$_y$ phase coexisted stably with them, indicating the Ni-Si alloy phase was formed during the nitridation process. In addition, with increasing the temperature from 1523 K to 1623 K, the oxygen partial pressure required to obtain Si$_3$N$_4$ gradually increased when the nitrogen partial pressure was 1 × 10$^{-1}$ MPa.

The macroscopic morphology of the products at 1250°C for 0 h nitridation were shown in Fig. 7a. Obviously, there was a layer of white-colored product above the sample doped with Ni additives, while this was almost absent in the sample without Ni. After the separation and testing of the white-colored product, it could be found that a large bulge peak appeared...
near $2\theta = 21^\circ$ in XRD pattern and its microstructure was fibrous and disordered, as shown in Fig. 7b and d, so it was mainly amorphous SiO$_2$ (JCPDS No.29–0085) nanowires caused by gas phase deposition. In addition, there were three Si diffraction peaks near $2\theta = 28.4^\circ$, 47.4$^\circ$ and 56.1$^\circ$, so the white-colored product also contained Si. This was due to partial disproportionation of SiO(g) at this temperature. These results indicate that Ni could promote the generation of SiO(g) at low temperature. But since O$_2$ was more active than N$_2$ at lower temperature, these SiO(g) was further oxidized by a small amount of O$_2$ in N$_2$ to form amorphous SiO$_2$ above the product.

In order to observe the effect of Ni on SiO$_2$ film more intuitively, the research ideas of surface physics and surface chemistry were introduced, and the equivalent substitution experiment was carried out by simulating a single DWSP particle with monocrystalline silicon block. The planar geometry of single-crystal silicon block was contributed to our study of the effect of Ni additives. As shown in Fig. 8a, when the single-crystal silicon block without Ni added on the surface was nitrided at 1250°C for 0 h, there was no obvious change on the silicon block surface, only some nanoparticles existed, which were SiO$_2$ nanoparticles detected by EDS (Fig. 8c). When the single-crystal silicon block with Ni added on the surface was nitrided at the same conditions, the surface crack occurred, as shown in Fig. 8b. And many fibrous products generated by gas reaction were found inside the crack, which were SiO$_2$ nanofibers detected by EDS (Fig. 8d). In addition, a small amount of Ni element was also detected inside the fracture. These results indicate that Ni could promote the generation of SiO(g) at lower temperature and destruct the SiO$_2$ film of the single-crystal silicon block surface.

Combined with the predominance area diagrams and the experimental results at 1250°C, the main reactions during nitridation are listed in Table 1. It could be seen that due to the high content of SiO$_2$ on the surface of the DWSP as a protective layer, Si did not react with N$_2$ during the initial nitridation stage, but reacted with SiO$_2$ first as shown in Eq. (1). The SiO$_2$ film cracked when the SiO partial pressure between Si and SiO$_2$ film was high enough. Then internal Si

Fig. 6 Si-O-N-Ni predominance area diagrams at different temperatures

Fig. 7 The macroscopic morphology (a) of the DWSP with and without Ni additives after nitridation at 1250°C for 0 h. Corresponding XRD pattern (b) and SEM image (c) of the white-colored product

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exposed in N₂ atmosphere and SiO(g) escaped. Due to the high partial pressure of oxygen at the initial stage and the high activity of O₂, a small amount of O₂ in N₂ will first participate in the reaction and react with SiO(g) and internal Si to form SiO₂, as shown in Eqs. (2, 3). As the partial pressure of oxygen gradually decreased, Si₃N₂O began to form through the Eq. (4). Then the unreacted SiO(g) would react with N₂ to form α-Si₃N₄ and O₂ through the Eq. (5), as shown in the rod-like products in Fig. 4. Meanwhile, the exposed internal Si would also react with N₂ to form Si₃N₄ through the Eq. (6), as shown in the granular products in Fig. 4. It could be found that when SiO(g) reacted with N₂ to form Si₃N₄, O₂ also was generated, which increased the partial oxygen pressure, and part of Si₃N₄ would also be oxidized into Si₂N₂O, as shown in Eq. (7). In addition, O₂ also reacted with unreacted Si and SiO(g) again. As the above process occurred repeatedly, a large number of rod-like products appeared.

Figure 9 illustrates the standard Gibbs free energy (ΔG⁰) for reactions (1)-(7). At a given temperature, the reaction proceeds spontaneously in the reverse direction when ΔG⁰ is greater than 0. On the contrary, the reaction can proceed spontaneously in the forward direction when ΔG⁰ is less than 0. The more negative the Gibbs free energy, the greater the tendency of the reaction. It can be found that the ΔG⁰ of reaction (1) is all greater than 0 at the experimental temperature, while the ΔG⁰ of reaction (5) also begins to be greater than 0 when

Table 1 Related chemical reactions in the nitridation of DWSP

| Chemical reactions | G°/kJ·mol⁻¹ | equation |
|--------------------|-------------|----------|
| Si(s)+SiO₂(g)=2SiO(g) | 714.21–0.360T | (1) |
| 2SiO(g)+O₂(g)=2SiO₂(s) | -1631.87+0.554T | (2) |
| Si(s)+O₂(g)=SiO₂(s) | -918.11+0.195T | (3) |
| 4Si(s)+2N₂(g)+O₂(g)=2Si₂N₂O(s) | -1827.38+0.557T | (4) |
| 6SiO(g)+4N₂(g)=2Si₃N₄(s)+3O₂(g) | -854.69+1.153T | (5) |
| 3Si(s)+2N₂(g)=Si₃N₄(s) | -733.87+0.330T | (6) |
| 4Si₃N₄(s)+3O₂(g)=6Si₁N₃O(s)+2N₂(g) | -2543.90+0.345T | (7) |

Fig. 9 The standard Gibbs free energy for reactions (1)-(7)
the temperature reaches above about 700°. The $\Delta G^\theta$ of other reactions are all less than 0 at the experimental temperature, indicating that all reactions can occur spontaneously except for reaction (1) and (5). It should be further noted that the $\Delta G^\theta$ of reactions (1) and (5) are also related to the partial pressure of SiO and the partial pressure of O$_2$, which can occur in actual nitridation [24].

Figure 10. illustrates the typical microscopic morphology and corresponding EDS results of the DWSP containing 2.0 wt% Ni after nitridation at 1300°C for 2 h. As shown in

![Fig. 11](image)

**Fig. 11** The schematic of the growth of Si$_3$N$_4$ during the nitridation of the DWSP doped with Ni
Fig. 10a, b and c, nanoparticles (red dotted circle) existed on the top of some \( \alpha\)-Si\(_3\)N\(_4\) nanorods. EDS (Fig. 10d) analysis showed that the top nanoparticles contained Si and Ni elements. Combined with the predominance area diagrams of Si-O-N-Ni system, nanoparticles existed on the top were Ni-Si eutectic liquid alloy. Based on previous research [25], vapor-liquid-solid (VLS) mechanism was suitable to describe the growth of nanorods with nanoparticles on top. First, Ni promoted the generation of SiO(g) at lower temperature and destruct the SiO\(_2\) film on the DWSP surface and then formed liquid Ni-Si alloy. Pigeon et al. pointed out that liquid phases were significant contributors to the production of Si vapor [26]. And since the solubility of gas in the liquid phase was higher than that in the solid state and the activation energy of liquid Si-N\(_2\) reaction was much lower than that required for the reaction of solid Si and N\(_2\) [27], Si vapor and N\(_2\) began to dissolve in large quantities in the liquid alloy and react after the formation of liquid Ni-Si alloy. \( \alpha\)-Si\(_3\)N\(_4\) began to precipitate when the liquid phase alloy reached a certain supersaturation. As the reaction gas continued to dissolve into the liquid alloy, \( \alpha\)-Si\(_3\)N\(_4\) continued to grow to form the morphology as shown in Fig. 10.

A schematic of the growth of Si\(_3\)N\(_4\) during the nitridation of the DWSP doped with Ni is shown in Fig. 11.

4 Conclusions

In this study, the DWSP doped with Ni additives were nitried at different temperatures. The results revealed that Ni markedly decreased the temperature at which the DWSP began to gain significant weight and extremely accelerated the nitridation of the DWSP at lower temperature. The DWSP containing 2.0 wt% Ni additives was completely nitried at 1300°C for 2 h, while the DWSP without Ni additives had not been nitried yet under the same conditions. Based on the equivalent substitution experiment of the single-crystal blocks, it could be concluded that Ni additives accelerated the nitridation and promoted the formation of the \( \alpha\)-Si\(_3\)N\(_4\) nanorods through facilitating the generation of the SiO(g) and destructing the SiO\(_2\) film on the surface of silicon at lower temperature. Meanwhile, Ni additives also played an important part in the growth of \( \alpha\)-Si\(_3\)N\(_4\) nanorods by forming liquid Ni-Si alloy in the product.

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Data Availability Not applicable.

Code Availability Not applicable.

Declarations

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