Recycling of the Diamond-wire Saw Powder by Ni-catalyzed Nitridation to Prepare Si_3N_4

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Research Article

Keywords: Diamond-wire saw powder, Ni catalyst, Nitridation, Catalytic mechanism, Silicon nitride

Posted Date: July 15th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-708745/v1>

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Abstract

In this paper, the acceleration of nickel (Ni) in 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 was 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 conducted that the presence of Ni additives accelerated the nitridation and promoted the formation of the α -Si₃N₄ 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 α -Si₃N₄ nanorods by forming liquid Ni-Si alloy in the product.

Introduction

Recently, the global energy crisis and environmental problems are becoming increasingly serious, due to the rapid growth of fossil energy consumption (Chen et al., 2016). So, it is imperative to find a new type of renewable energy, in which solar energy is emerging and promising (Liu et al., 2019b; Wu et al., 2017). Currently, the solar cells used in the photovoltaic industry are mainly crystalline silicon solar cells, which account for about 90% (Chen et al., 2018). In the production of crystalline silicon wafers, diamond-wire saw technology possessed great advantages and development potential (Cao et al., 2015). Nevertheless, by reason of the diameter of diamond wire was close to the thickness of the silicon wafer, nearly 40% of the diamond-wire saw powder was produced and thrown away as waste during the silicon ingot slicing process (Maeda et al., 2014). The DWSP mainly contains Si and SiO₂, with a small amount of metal impurities, such as Ca, Al, Fe, Ni and so on (Yang et al., 2019a; Yang et al., 2019c). The particle size of the DWSP was very fine (~ 1µm), which belongs to PM 2.5 pollutants. It will cause secondary pollution to the soil, air and water resources, resulting in safety and environment problems. Therefore, corresponding treatment is extremely required to recycle it.

To solve the problems above mentioned, a lot of efforts on the recycle of high-purity silicon had been carried out (De Sousa et al., 2016; Kong et al., 2019; Lu et al., 2019; Tomono et al., 2013; Yang et al., 2019b). Although these studies had yielded remarkable results, there is still a long way to go for using as solar-grade feedstocks. 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 (Liu et al., 2019a; Yang et al., 2019a). 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 (Hardie and Jack, 1957; Long et al., 2018). At present, the most commonly method to prepare

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trial silicon powder (Liu et al., 2017). By contrast, 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. Therefore, some researches on the rapid nitridation of the DWSP at low temperature had been carried out, one of which is to accelerate the nitridation of the DWSP by using metal additives (Gu et al., 2019; Lan et al., 2020; Luo et al., 2020). Nevertheless, to our knowledge, researches on the nitridation of the DWSP at low temperature by the metal additives promoted is quite small. Thus, the mechanism by which metal additives promote the nitridation of the DWSP is still unclear.

Considering that the DWSP contains a small amount of metal impurities, if these metals can accelerate the nitridation process at low temperature, it will be of great significance for the recycling of the DWSP and energy saving. 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 through the nitridation of single-crystal blocks instead of the DWSP. This work provides a scientific basis for the preparation of silicon nitride at low temperature by the nitridation of diamond-wire saw powder.

Materials And Methods

2.1 Raw material

The DWSP (Henan Xin Da New Material Co., LTD, China) and nickel powders (99.9% pure, $\leq 100\text{nm}$, 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 N_2 (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 ($45\times 22\text{mm}$) and then put it into the constant temperature zone of a tube furnace. Before heating, purifying the tube furnace with nitrogen flow for 30 minutes 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.

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 was analyzed by FactSage 7.2. X-ray diffraction (XRD; D8 Advance, Loading [MathJax]/jax/output/CommonHTML/jax.js) 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.

Results And Discussion

3.1 Thermogravimetric analysis

The nitridation behavior of the DWSP before and after doping with Ni was studied through thermogravimetric analysis in the nitrogen atmosphere, the test temperature rose from 25°C to 1460°C at a heating rate of 10°C/min, as shown in Fig. 1. It could be found that the variation trend of both was similar, but the mass gain of the DWSP doped with Ni was obviously higher than that of the direct nitridation of the DWSP. In addition, both TG curves had obvious inflection points, when the test temperature exceeded the temperature of the inflection point, the mass began to increase significantly. 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 significant weight.

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 peak was detected, indicating the DWSP had not been nitrided at this temperature evidently. On increasing the temperature to 1350°C, Si_3N_4 and tiny $\text{Si}_2\text{N}_2\text{O}$ peaks were detected, but unreacted Si peaks maintained a high intensity, indicating the nitridation extent of the DWSP was still low. With increasing the temperature to 1400°C, the intensity of Si_3N_4 peaks increased evidently, which indicates that the DWSP without Ni additives could effectively form Si_3N_4 only when the temperature reached 1400°C. Upon 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 Si_3N_4 formed at 1300°C, and only 56.1 wt.% of Si_3N_4 was formed at 1350°C. When improving the temperature from 1350°C to 1400°C, the content of Si_3N_4 increased to 80.5% 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, Si_3N_4 and a small amount of $\text{Si}_2\text{N}_2\text{O}$ 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, $\alpha\text{-Si}_3\text{N}_4$, $\beta\text{-Si}_3\text{N}_4$ and $\text{Si}_2\text{N}_2\text{O}$ 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, $\text{Si}_2\text{N}_2\text{O}$ was detected in the samples whether it doped with Ni additives or not, there were two reasons: one was the high content of SiO_2 in the DWSP (about 17 wt.%) (Jin et al., 2019) and the other was the trace oxygen contained in high-purity nitrogen.

Figure 4 presents typical SEM images and corresponding EDS results of the samples doped with 0–2.0 wt.% Ni after nitridation at 1300°C for 2 h. 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. And 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 1.0wt.% 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 (Huang et al., 2014; Longland and Moulson, 1978) these granular products were Si_3N_4 formed by gas-solid reaction and these nanorods were $\alpha\text{-Si}_3\text{N}_4$ 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 prduct and nanorods except Si and N elements by EDS (Fig. 4f and i). It could be attributed to the surface of Si_3N_4 was oxidized into $\text{Si}_2\text{N}_2\text{O}$ and the presence of SiO_2 amorphous layer (Wang et al., 1998; Zhang et al., 2001).

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_3N_4 and $\text{Si}_2\text{N}_2\text{O}$ peaks appeared. On increasing the temperature to 1250°C, the intensity of Si_3N_4 and $\text{Si}_2\text{N}_2\text{O}$ 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, $\text{Si}_2\text{N}_2\text{O}$ 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_3N_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 $\text{Si}_2\text{N}_2\text{O}$ decreased from 19.6 wt.% to 5.9 wt.%, it could be attributed to the decomposition of $\text{Si}_2\text{N}_2\text{O}$ (Jin et al., 2019a).

3.5 Analysis of Ni accelerated nitridation mechanism

The predominance area diagrams of Si-O-N-Ni system at 1523K and 1623K 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_3N_4 and $\text{Si}_2\text{N}_2\text{O}$ in the equilibrium state, so there would be no Si_3N_4 and $\text{Si}_2\text{N}_2\text{O}$ 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 $P(\text{N}_2)$ of 1×10^{-1} MPa, different oxygen partial pressures correspond to different products. As the partial pressure of oxygen increased, Si_3N_4 was gradually oxidized into $\text{Si}_2\text{N}_2\text{O}$ and eventually into SiO_2 . And it could be found that in the stable regions of $\text{Si}_2\text{N}_2\text{O}$ and Si_3N_4 , Ni_xSi_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 1523K to 1623K, the oxygen partial pressure required to obtain Si_3N_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 the sample without Ni almost did not have this phenomenon. After separating and testing, it could be found

Loading [MathJax]/jax/output/CommonHTML/jax.js = 21° in XRD pattern and the microstructure of the white-

colored was fibrous and disordered, as shown in Fig. 7b and d, so it was mainly amorphous SiO_2 nanowires caused by gas phase deposition. In addition, there were three Si diffraction peaks around $2\theta = 28.4^\circ$, 47.4° and 56.1° , so the white-colored product also contained Si. This was due to the disproportionation reaction of partial 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 nitriding 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 exposed in N_2 atmosphere and SiO(g) escaped. Due to the high partial pressure of oxygen at the initial stage and the high activity of O_2 , a small amount of O_2 in N_2 will first participate in the reaction and react with SiO(g) and internal Si to form SiO_2 , as shown in Eq. (2, 3). As the partial pressure of oxygen gradually decreased, $\text{Si}_2\text{N}_2\text{O}$ began to form through the Eq. (4). Then the unreacted SiO(g) would react with N_2 to form $\alpha\text{-Si}_3\text{N}_4$ and O_2 through the Eq. (5), as shown in the rod-like products in Fig. 4. Meanwhile, the exposed internal Si would also react with N_2 to form Si_3N_4 through the Eq. (6), as shown in the granular products in Fig. 4. It could be found that when SiO(g) reacted with N_2 to form Si_3N_4 , O_2 also was generated, which increased the partial oxygen pressure, and part of Si_3N_4 would also be oxidized into $\text{Si}_2\text{N}_2\text{O}$, as shown in Eq. (7). In addition, O_2 also reacted with unreacted Si and SiO(g) again. As the above process occurred repeatedly, a large number of rod-like products appeared.

Table 1
Related chemical reactions in the nitridation of DWSP.

Chemical reactions	$\Delta G^\theta/\text{kJ}\cdot\text{mol}^{-1}$	equation
$\text{Si(s)} + \text{SiO}_2\text{(s)} = 2\text{SiO(g)}$	$714.21 - 0.360T$	(1)
$2\text{SiO(g)} + \text{O}_2\text{(g)} = 2\text{SiO}_2\text{(s)}$	$-1631.87 + 0.554T$	(2)
$\text{Si(s)} + \text{O}_2\text{(g)} = \text{SiO}_2\text{(s)}$	$-918.11 + 0.195T$	(3)
$4\text{Si(s)} + 2\text{N}_2\text{(g)} + \text{O}_2\text{(g)} = 2\text{Si}_2\text{N}_2\text{O(s)}$	$-1827.38 + 0.557T$	(4)
$6\text{SiO(g)} + 4\text{N}_2\text{(g)} = 2\text{Si}_3\text{N}_4\text{(s)} + 3\text{O}_2\text{(g)}$	$-854.69 + 1.153T$	(5)
$3\text{Si(s)} + 2\text{N}_2\text{(g)} = \text{Si}_3\text{N}_4\text{(s)}$	$-733.87 + 0.330T$	(6)
$4\text{Si}_3\text{N}_4\text{(s)} + 3\text{O}_2\text{(g)} = 6\text{Si}_2\text{N}_2\text{O(s)} + 2\text{N}_2\text{(g)}$	$-2543.90 + 0.345T$	(7)

Figure 9. illustrates the standard Gibbs free energy for reactions (1)-(7). It can be found that the ΔG^θ of reaction (1) is all greater than 0 at the experimental temperature, while the ΔG^θ of reaction (5) also begins to be greater than 0 when the temperature reaches above about 700°. The ΔG^θ 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 ΔG^θ of reactions (1) and (5) are also related to the partial pressure of SiO and the partial pressure of O₂, which can occur in actual nitridation ((Jin et al., 2019b)).

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. 10a, b and c, nanoparticles (red dotted circle) existed on the top of some $\alpha\text{-Si}_3\text{N}_4$ 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 (Wagner and Ellis, 1964), 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 SiO₂ 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 (Pigeon et al., 1993). 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-N₂ reaction was much lower than that required for the reaction of solid Si and N₂ (Mukerji and Biswas, 1981), Si vapor and N₂ began to dissolve in large quantities in the liquid alloy and react after the formation of liquid Ni-Si alloy. $\alpha\text{-Si}_3\text{N}_4$ began to precipitate when the liquid phase alloy reached a certain supersaturation. As the reaction gas continued to dissolve into the liquid alloy, $\alpha\text{-Si}_3\text{N}_4$ continued to grow to form the morphology as shown in Fig. 10.

A schematic of the growth of Si_3N_4 during the nitridation of the DWSP doped with Ni is shown in Fig. 11.

Conclusions

In this study, the DWSP doped with Ni additives were nitrided 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 nitrided at 1300°C for 2 h, while the DWSP without Ni additives had not been nitrided yet under the same conditions. Based on the equivalent substitution experiment of the single-crystal blocks, it could be conducted that 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 $\text{SiO}(\text{g})$ 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 $\alpha\text{-Si}_3\text{N}_4$ nanorods by forming liquid Ni-Si alloy in the product.

Declarations

Acknowledgments

This work was supported by the National Natural Science Foundation of China (Grant No. 21978045).

Funding: This work was supported by the National Natural Science Foundation of China (Grant No. 21978045).

Conflicts of interest/Competing interests: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Availability of data and material: Not applicable.

Code availability: Not applicable.

Authors' contributions: All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Hongli Xu and Xing Jin. The first draft of the manuscript was written by Hongli Xu and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Compliance with Ethical Standards

Conflict of Interest: The authors declared that they have no conflicts of interest to this work.

Consent to Participate: Not Applicable.

Consent for Publication: Written informed consent for publication was obtained from all participants.

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Figures

Figure 1

TG curves of the DWSP with and without Ni additives

Figure 2

XRD patterns (a) and relative contents of the sample phases (b) of the DWSP without Ni additives after nitridation at different temperatures for 2 h.

Figure 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.

Figure 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.

Figure 5

XRD patterns (a) and relative contents of the sample phases (b) of the DWSP doped with 2.0 wt.% Ni additives after nitridation at different temperatures for 2 h.

Figure 6

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

Figure 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.

Figure 8

SEM images of the surface of the single-crystal silicon block after nitridation at 1250°C for 0 h: (a) without Ni, (b) with Ni. And EDS results (c and d) of point A and area B.

Figure 9

The standard Gibbs free energy for reactions (1)-(7).

Figure 10

Typical SEM images (a, b and c) of the DWSP doped with 2.0 wt.% Ni after nitridation at 1300°C for 2 h and corresponding EDS results (d) of point A.

Figure 11

The schematic of the growth of Si_3N_4 during the nitridation of the DWSP doped with Ni.