Preparation of irregular silica nano-abrasives for the chemical mechanical polishing behaviour on sapphire substrates

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Abrasives directly affect the polishing rate and surface quality for sapphire chemical mechanical polishing (CMP). In this work, irregular silica nano-abrasives were synthesised by nickel ion-induced effect combined with growth method in order to simultaneously acquire a high polishing rate and a smooth and flat surface quality on sapphire. The synthesis process of irregular silica nano-abrasives is predicted and analysed by zeta potential. The irregular silica nano-abrasives have a distinguished CMP behaviour for a 22.72% increase in polishing rate and a flat surface quality of sapphire. X-ray photoelectron spectroscopy analysis results have confirmed these solid-phase chemical reactions happen during the sapphire CMP process. In addition, it was analysed about the polishing process and mechanism by establishing a contact model between nano-abrasives and sapphire.

1. Introduction: Sapphire (α-Al_2O_3) is a kind of material that combines mechanical properties, optical properties, thermal properties, dielectric properties, physical properties and chemical properties [1–5]. As a result, a large number of sapphires are used in many fields such as industrial, communications, defence, as infrared window, semiconductor chip, fingerprint recognition and other materials [6]. At present, the application of sapphire, especially in optoelectronic devices, has increased dramatically. Sapphire is used as a substrate material on light emitting diodes [7–9], which is recognised as one of the most promising high-technology fields in the 21st century. This puts higher demands on the surface quality of sapphire. A smooth and clean surface is not only the need to beautify the product, but also a critical factor in extending workpiece life and ensuring product quality. Unfortunately, there has always been a challenging problem in this domain, i.e. the high hardness and high chemical stability of sapphire [10, 11].

To flatten the surface of sapphire without destroying the original properties, chemical mechanical polishing (CMP) is an appropriate choice [12]. CMP combines mechanical action of nano-abrasives with chemical action of nano-abrasives to deal with workpieces, thereby obtaining ultra-flat and ultra-smooth surface, which can achieve a dynamic balance that allows the plane of the sapphire substrate to reach atomic levels [4, 13]. It is the only technology that enables to provide a global flattening and is widely used in many fields [14].

Abrasives play an important role in the CMP process of sapphire, which is an important implementer and deliver of mechanical action and chemical action, and whose type, shape, structure, hardness, particle size and other properties of the abrasives all affect the polishing behaviour [15, 16]. Thus, nano-abrasives are continuously studied to make material removal rate (MRR) of CMP increase, and make the surface quality of the workpiece improve. Silicon oxide is usually used in the sapphire CMP process in the light of its good dispersibility, stability, easy cleaning, easy storage and low cost. Bun-Athuek *et al.* [9] analysed the effects of two different size colloidal silica abrasives (20 and 55 nm) on sapphire surface during CMP. In order to improve MRR of sapphire and acquire an outstanding surface, Liu and Lei prepared a core–shell structure of Sm^{3+}-doped colloidal silica abrasives on the CMP of sapphire [17]. Amorphous silica is a small particle with a nanometre size and therefore has a much lower microscratch and defect rate. However, the MRR of silica nano-abrasives (that is always spherical and soft in nature) is relatively low. This problem has led to more attention of scientists.

Hence, the preparation of irregular silica nano-abrasives (ISNAs) for CMP on sapphire is proposed. Recently, a few researchers have prepared non-spherical silica nano-abrasives. Liang's Group synthesised non-spherical silica nano-abrasives from $Zn(Ac)_2$ as cation source and tetraethoxysilane (TEOS) as raw material [18], and discussed the effects of CMP on silica wafers. Apparently, TEOS is not environmentally friendly. Lee *et al.* [19] also synthesised ISNAs by way of the multi-step method. The MRR of SiO_2 film CMP is improved. Even so, there is no complete theory on the material removal mechanism, which needs to further study.

In this work, irregular (potato-shaped, chain-shaped, flower-shaped etc.) silica nano-abrasives were synthesised by the nickel ion-induced effect combined with growth method to make the MRR increase, and make the surface roughness (Sa) reduce on sapphire. The synthesis process of the ISNAs was analysed and a model was established to discuss the material removal mechanism.

2. Experimental section

2.1. Materials: Nickel nitrate hexahydrate [$Ni(NO_3)_2 \cdot 6H_2O$, AR, Aladdin Chemical Regent Co., LTD, China]; sodium hydroxide (NaOH, AR, Sinopharm Chemical Reagent Co., LTD, China); hydrochloric acid (HCl, GR, Sinopharm Chemical Reagent Co., LTD, China); sodium silicate (Na_2SiO_3, JiNan DeWang Chemical Industry Co., LTD, China); cation exchange resin (Sinopharm Chemical Reagent Co., LTD, China) are used.

Unpolished sapphire with the *c* (001) orientation of φ 50.8 mm × 0.45 mm (Jiangsu HaoHan Sapphire Science and Technology Co., LTD, China); Rodel porous polyurethane polishing pad; UNIPOL-1502 CMP equipment (Shenyang Kejing instrument, Co. LTD, China) are used.

2.2. Preparation process: ISNAs were synthesised by the nickel ion-induced effect combined with growth method. The specific process is as follows: first of all, active silicic acid is prepared by ion exchange method [14, 20]. Next, activated silicic acid that

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has been prepared was placed into a four-necked flask. At the same time, the nickel nitrate solution was slowly added drop by drop. In addition, the flask was heated and allowed to boil for 10 min. Then, the active silicic acid solution was dropped through a constant pressure separating funnel to make silica nano-abrasives grow with the pH = 10 adjusted by sodium hydroxide solution during the experimental process. At last, the ISNAs can be obtained.

Spherical silica nano-abrasives (SSNAs) were synthesised directly via silica seed growth method [14], in which the silica seed was firstly put in a flask. Nickel nitrate solution and active silicic acid solution are added to grow silica nano-abrasives. Then, SSNAs were synthesised. The pure and spherical silica nano-abrasives (PSNAs) with the nickel compounds content 0 wt.% is synthesised the same as the ISNAs, except that no nickel compounds are added.

In our work, ISNAs were synthesised. The polishing liquid was obtained with the content of 10 wt.% on silica nano-abrasives.

2.3. Polishing tests: Polishing liquid was come from the ISNAs with a total solid content of 10 wt%. The relative parameters were that the pressure of 6 kg, the plate rotating speed of 70 rpm and the polishing liquid supplying rate of 530 ml min^{-1} in polishing tests. CMP behaviour was monitored.

The MRR (μm h^{-1}) is calculated by

$$\mathrm{MRR} = \frac{\Delta m \times 10^6}{\rho \pi R^2 \tau} \tag{1}$$

where Δm (g) is the weight difference of before and after polishing for sapphire; ρ (g cm^{-3}) is the density of sapphire, $\rho_{\mathrm{sapphire}} =$ 3.98 g cm^{-3}; R (nm) is the radius of sapphire substrates; τ (h) is the polishing time, $\tau = 2$ h.

2.4. Characterisations: The morphology of ISNAs was detected through scanning electron microscopy (SEM, JSM-7500F) with a voltage of 15 kV.

Zeta potential of ISNAs was measured by Zetasizer 3000HS (Malvern Instruments Ltd., UK).

The surface roughness (Sa, which is mean deviation) of sapphire was analysed through Ambios Xi-100 surface profiler (Ambios Technology Corp., USA).

Elemental composition in polishing liquid was identified through X-ray photoelectron spectroscopy (XPS, ESCALAB250Xi) with an excitation source Al K_α radiation. The reference was C1s with a binding energy of 284.6 eV.

3. Results and discussion

3.1. Morphologies analysis: Fig. 1 shows the SEM images of SSNAs and ISNAs with the nickel compounds content of 0.8 wt.%, where the SSNAs have good dispersibility and a relatively uniform shape, and the size is about 45 nm, and the ISNAs had a variety of shapes, such as potato-shaped, chain-shaped, flower-shaped and so on. This could mean ISNAs have been synthesised via nickel ion-induced effect combined with growth method.

3.2. Synthesis process of ISNAs: To study the preparation mechanism on ISNAs, zeta potential of silica nano-abrasives was measured. Fig. 2 shows the change of zeta potential of ISNAs with the contents of nickel compounds, which is the same as expected [14]. The absolute value of the potential does drop gradually as nickel compound content increases, which can significantly cause the electrostatic repulsion between the silica nano-abrasives to decrease, which would destroy the stable normal state of the silica nano-abrasives [21]. When heat is supplied to the small nano-particles, the small nano-particles are closer or even get together at the initial stage of formation via two or more particles due to the thermal motion to colliding with each other and the strong attraction with each other.

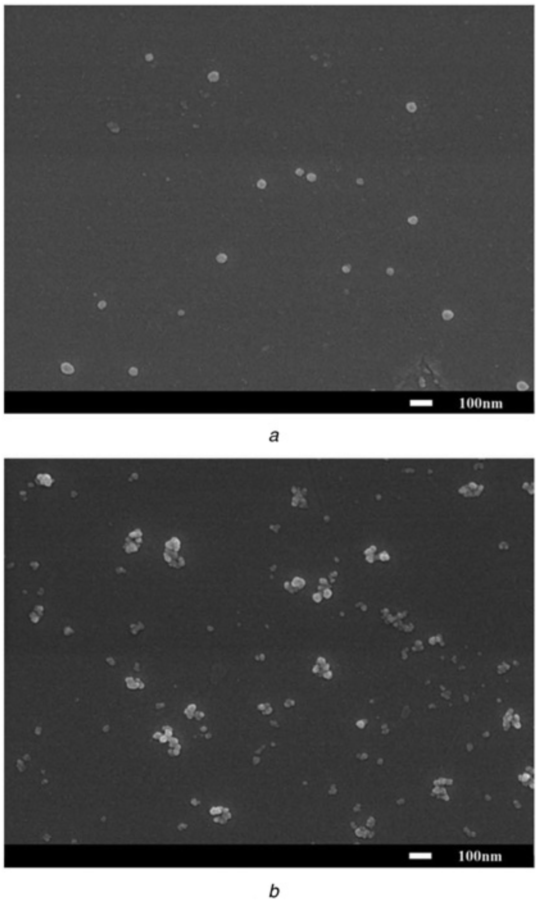

Fig. 1 *SEM images of silica nano-abrasives*
a SSNAs
b ISNAs

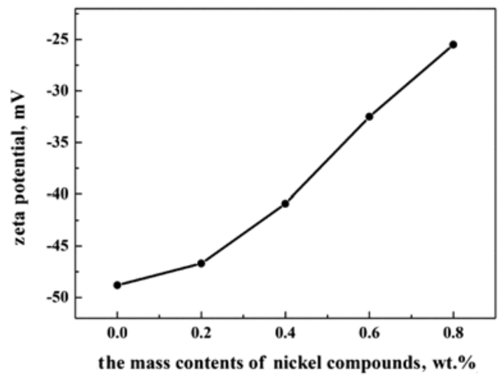

Fig. 2 *Zeta potential of ISNAs as the contents of nickel compounds (wt.%)*

However, the addition of nickel compounds makes the stability of silica nano-abrasives affected. According to Derjguin-Landau-Verwey-Overbeek (DVLO) theory [22], the reason why the silica nano-abrasives maintain a good stability is because the electrostatic repulsion is greater than Van der Waals forces. That is to say, the content of nickel compounds plays an important role in the stability on polishing fluid. When the content of the nickel compound exceeds a certain amount, the zeta potential will excessively decrease, thereby being insufficient to resist Van der Waals force among particles [14]. The silica may agglomerate or even precipitate, resulting in scratches and damages for the surface of sapphire. Therefore, the amount of Nickel compounds should be well controlled to ensure the stability of the system.

Based on the above analysis, the synthesis process of ISNAs is speculated. Tai showed that in the slightly acidic, neutral and slightly alkaline solution [23], silicic acid exists in two forms of $H_3SiO_4^-$ and H_4SiO_4. When they meet, the polymerisation reaction can occur immediately. The reaction product can be further reacted with $H_3SiO_4^-$ to form silicic acid polymer, which is further polymerised to form silica. At the beginning, the addition of the nickel compound compresses the electric double layer of the silica small particles so that the electrostatic interaction among the particles is reduced, causing the silica small particles to grow unevenly. The nickel compound and silica small particles may

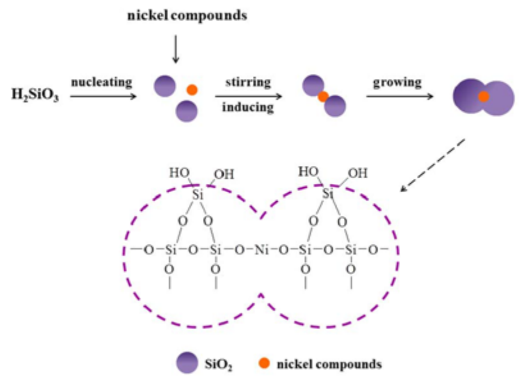

Fig. 3 *Schematic diagram of synthesis process for ISNAs*

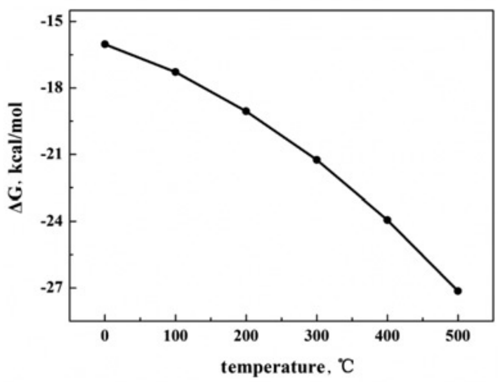

Fig. 4 *Relationship between the Gibbs free energy and temperature for Ni(OH)$_2$ and SiO$_2$*

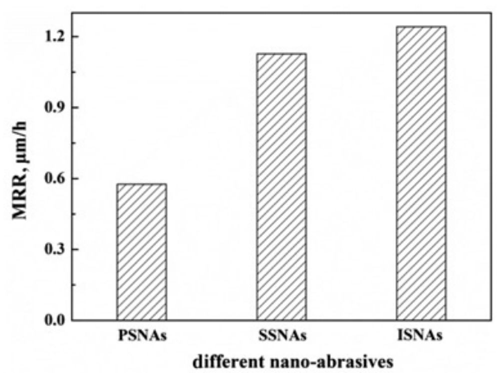

Fig. 5 *MRRs of PSNAs, SSNAs and ISNAs (the nickel compounds content of 0.6 wt.%)*

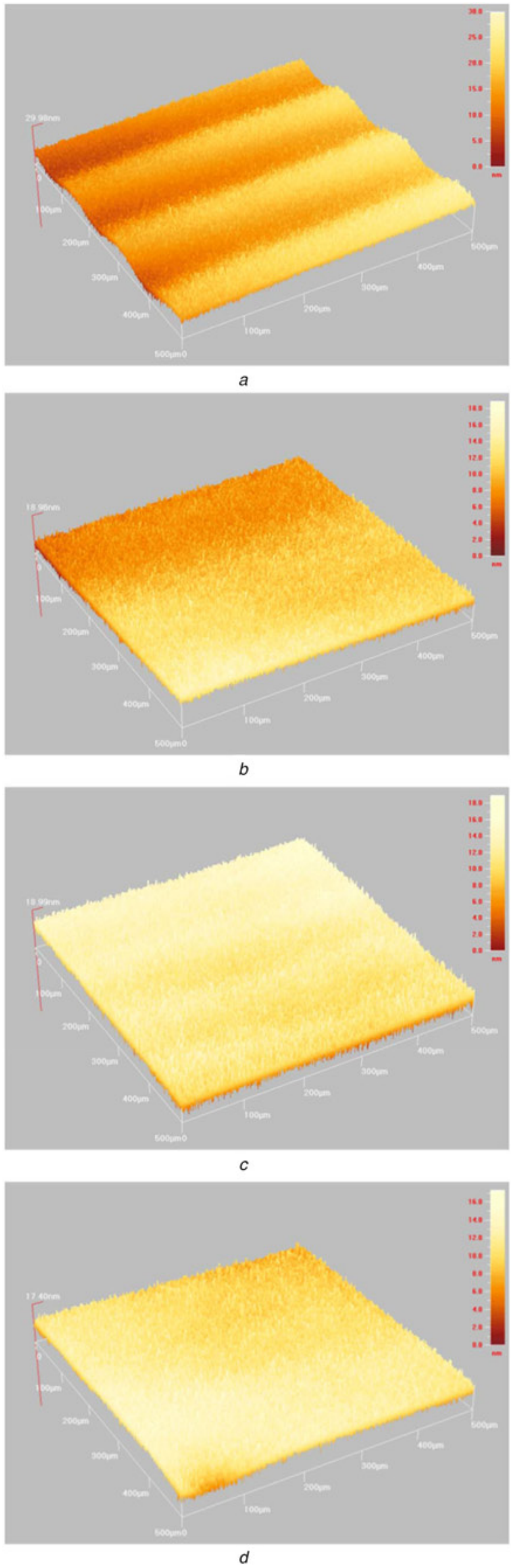

Fig. 6 *Surface profiles of sapphire (the nickel compounds content of 0.6 wt.%)*
a Before polishing
b Polished by PSNAs
c Polished by SSNAs
d Polished by ISNAs

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react as shown in reaction (2), where Ni_2SiO_4 is formed, containing –Si–O–Ni–O–Si– [14, 24]. Then, the above product is further polymerised and grown. Finally, ISNAs are synthesised. The predicted synthesis process is shown in Fig. 3

$$2Ni(OH)_2 + SiO_2 \rightarrow Ni_2SiO_4 + 2H_2O \quad (2)$$

For the purpose of further understanding the possibility of the presence of Ni_2SiO_4, HSC Chemistry software was used to trace and analyse. The result in Fig. 4 shows the Gibbs free energy (G) is less than zero between 0 and 500°C. Equation (2) can be carried out when the temperature was 100°C in the experiment. This further indicates the feasibility of the reaction.

3.3. CMP behaviour on sapphire substrates of ISNAs: Generally, two parameters were used to discuss the sapphire polishing behaviour, where two parameters are MRR and Sa.

Fig. 5 is the MRRs of PSNAs, SSNAs and ISNAs. It can be clearly found that the MRR of PSNAs is the lowest, the MRR of SSNAs is second and ISNAs has a highest MRR. At the same time, Fig. 6*a* is the unpolished surface profiles and Figs. 6*b*–*d* are the surface profiles polished by PSNAs, SSNAs and ISNAs. The unpolished sapphire has a rough surface (Sa = 3.531 nm), but the Sa of sapphire after polishing is greatly decreased. The Sa of 2.002, 1.786 and 1.678 nm is polished by the pure silica nano-abrasive, the SSNA and the ISNA. This can be clearly noticed that the surface of sapphire polished by ISNA has been improved compared to that polished by the pure silica nano-abrasive and the SSNA.

The MRR and Sa of spherical and ISNAs with the different content of nickel compounds are explored as shown in Fig. 7. Obviously, the MRR of ISNAs is higher compared to that of SSNAs in Fig. 7*a*. The MRR of SSNAs increases as the content of nickel compounds increases, but the MRR is almost unchanged with the content of nickel compounds attained at 0.8 wt.%. This may be that a little precipitation in the polishing liquid is found clearly owing to the excessive addition of the nickel compounds, which causes the particles to precipitate. Large particles would cause damages to the surface of sapphire. However, the MRR of ISNAs gradually increases as the content of nickel compounds increases. The MRR of the ISNAs is 1.383 µm/h, which is 22.72% higher than that of the SSNAs at the optimal mass content. Fig. 7*b* reveals the Sa of sapphire polished by s SSNAs and ISNAs with the different contents of nickel compounds. As can be seen, the 0.8 wt.% is an optimal mass content, where the Sa is lower with 1.593 nm. This means an outstanding combination and a balance point so that a high MRR can be achieved without damaging surface roughness.

3.4. Material removal mechanism: Sapphire CMP is a process that has been studied for a long time and is being explored in depth. Both chemical effect and mechanical effect are very essential for CMP. In recent decades, the material removal mechanism is discussed and explored.

XPS has been carried out so as to know the existing form of elements. Fig. 8 displays the XPS spectra of elements on ISNAs

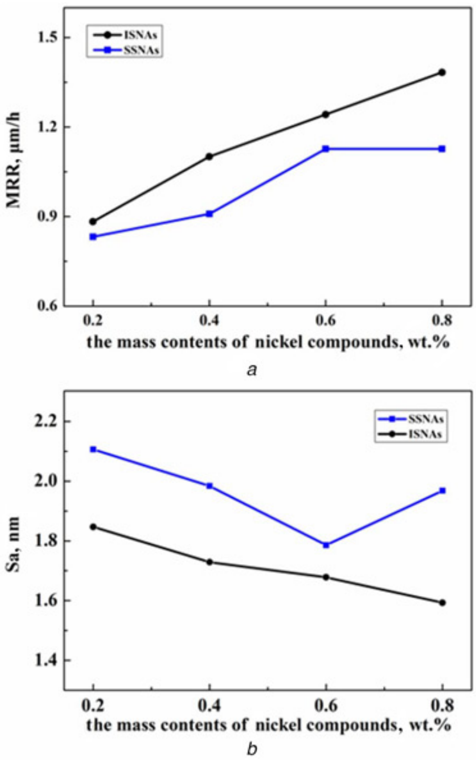

Fig. 7 *CMP behaviour of different nickel compounds contents (wt.%) on sapphire*
a MRR
b Sa

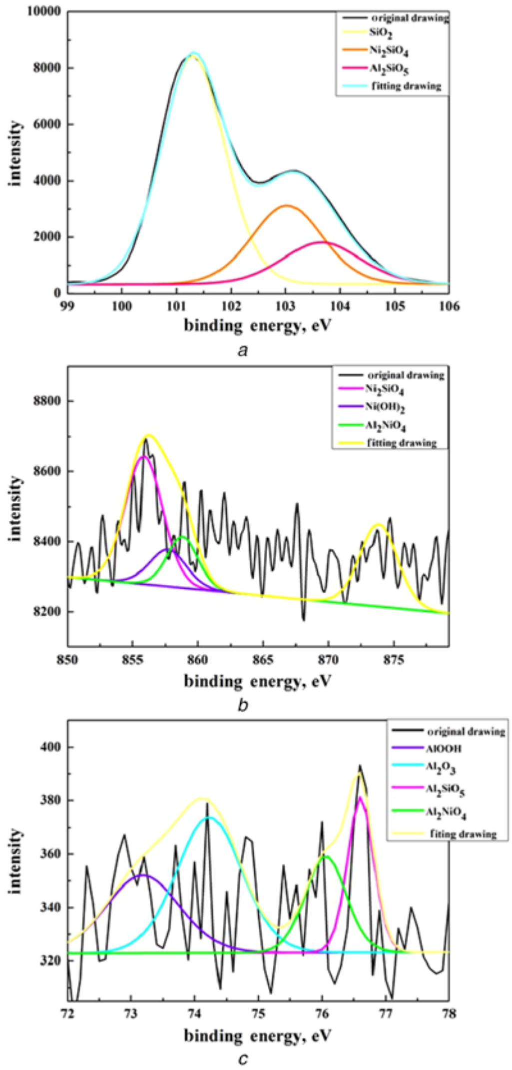

Fig. 8 *XPS spectra of elements from ISNAs after polishing*
a Si 2p
b Ni 2p
c Al 2p

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Table 1 Binding energy analysis of elements

Elements	Si 2p	Ni 2p	Al 2p
binding energy, eV	101.3	855.8	73.2
	103.0	858.8	74.2
	103.7	857.6	76.1
		873.8	76.6

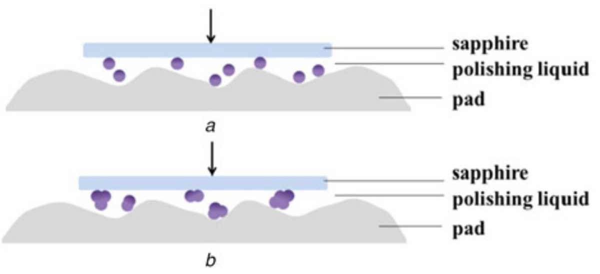

Fig. 9 *Schematic diagram of contact model*
a SSNAs
b ISNAs

after polishing. Binding energy corresponding to the peaks has been described in Table 1. It is clearly noted that Si 2p has three chemical states, and Ni 2p also has three chemical states with a double peak. In addition, there are four chemical states for Al 2p, where it implies that there are four compounds. The analysis results have confirmed that solid-phase chemical reactions happen as shown in the following chemical reactions [5, 14, 25]:

$$Al_2O_3 + H_2O \rightarrow 2AlOOH \quad (3)$$

$$SiO_2 + 2AlOOH \rightarrow Al_2SiO_5 + H_2O \quad (4)$$

$$Ni(OH)_2 + Al_2O_3 \rightarrow NiAl_2O_4 + H_2O \quad (5)$$

Lei and Gu [5] and Vovk *et al.* [25] also have shown that solid-phase chemical reactions have been confirmed between sapphire and silica nano-abrasives. Chemical action is important, but the mechanical effect is not to be underestimated. Luo and Dornfeld [26] proposed the elastic contact assumption among wafer, abrasive and pad. They improved the formula for the MRR, even they pointed out that the MRR will increase as the contact area increases. Sun *et al.* [27] have studied that the particles can be easy to slide motion when the irregularity of particles is increased. The proportion of slide motion of the particles will increase reaching a certain level, and the final expression is the ascension on the coefficient of friction, leading to increasing in mechanical action and material removal.

What is more, a contact model was established with respect to nano-abrasives and sapphire in Fig. 9, where the contact area of the ISNAs with sapphire is greatly increased compared with the SSNAs. More solid-phase chemical reactions will occur as the contact area increases. At the same time, the mechanical effect is enhanced as the contact area increases based on Luo's theory [26]. The larger the contact area, the greater the MRR. It can be found apparently that the form of motion of the SSNA and ISNAs is different. Fig. 9 also shows that the SSNA is more prone to rolling motion, but the ISNA is more inclined to a unique motion that is sliding motion [28]. This will increase the coefficient of friction between nano-abrasives and sapphire. The ISNA has an approximately spherical shape with a smooth surface, and the silica nano-abrasives itself has a low hardness, resulting in the surface roughness of sapphire not sacrificed. It means plough effect will become weak because of the addition of the contact area between sapphire and ISNAs. There is not significantly negative influence for the Sa so that the surface quality of sapphire is good. This is consistent with our CMP tests results with an excellent CMP behaviour on sapphire.

All in all, the organic combination of mechanical action and chemical action reaches an equilibrium when the content of nickel compound is 0.8 wt.%.

4. Conclusions: The purpose of this research is to prepare ISNAs and study its CMP behaviour on sapphire. After a series of experiments, the main conclusions are as follows: firstly, ISNAs were synthesised by nickel ion-induced effect combined with growth method. Secondly, the structure of ISNAs can contain Ni_2SiO_4 and –Si–O–Ni–O–Si– bond, and the existence of Ni_2SiO_4 is confirmed through HSC Chemistry software. Thirdly, ISNAs have an outstanding CMP behaviour. This is because the contact area between ISNAs with sapphire increases compared with SSNAs, which results in more solid-phase chemical reactions to occur. In addition, the ISNAs tend to sliding motion due to its unique shape, which also lead to the ascension about MRR. What is more, the surface quality on sapphire also has been improved. The study would have a potentially promising application prospect on sapphire.

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