

Crystal Phases and Chemical Stabilities of YSi_2 Powders Fabricated from Low and High Purity Si and Y Powders

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Abstract

Y-Si compounds with the composition of Y:Si = 1:2 were fabricated using Yttrium and Silicon raw powders with low and high purity in various atmospheres and temperatures. Although the latest Y-Si phase diagram shows that the α - and β - YSi_2 phases are the stable phases for the stoichiometric composition of Y:Si = 1:2, the current experimental results suggest that the high temperature phase with the hexagonal structure, β - Y_3Si_5 , would be the stable phase for this composition, and that the high temperature phase with the orthorhombic structure, β - YSi_2 , would be the meta-stable phase with high oxygen impurity content. It was demonstrated that YSi_2 powders possess much superior chemical stability than Yttrium metal. It was found that the best dispersing solvent was 2-propanol for YSi_2 powder.

Keywords

YSi_2 , Yttrium Silicide, Crystal Phase, Chemical Stability, Oxygen Impurity

1. Introduction

The latest Y-Si phase diagram shown in **Figure 1** [1] demonstrated four congruently melting compounds, Y_5Si_3 (melting point (m.p.) 1844°C), Y_3Si_4 (m.p. 1860°C), YSi (m.p. 1878°C) and Y_3Si_5 (m.p. 1612°C). In addition to these phases, there is one peritectically melting compound, YSi_2 (m.p. 1508°C). The phase diagram [1] was calculated based on thermodynamic data [2], however the polymorphic transformations in Y_3Si_5 and YSi_2 were not based on thermodynamic calculation but on the thermodynamic assessments [2] [3]. Among, these five compounds, the existence of the YSi_2 phase had been controversial. A detailed investigation of phase equilibria and crystal structures of the compounds by

methods of thermal, metallographic, x-ray phase analyses and by hardness measurement was carried out [4]. The existence of the compounds Y_5Si_3 , Y_5Si_4 , YSi , and Y_3Si_5 was established, but the formation of YSi_2 was not confirmed as a stable phase. Thus, the former version of the Y-Si phase diagram [5] did not include the YSi_2 phase. Later, the method of measuring the electromotive forces (*emf*) of the concentration galvanic elements determined the thermodynamic functions of the formation of the Y-Si compounds, and all five compounds were confirmed [6]. The standard enthalpies of formation for some 4d transition metal silicides have been measured by high temperature direct synthesis calorimetry at 1473 K, and the YSi_2 was also confirmed as a stable phase [7].

Early works regarding rare-earth disilicides [8] [9] [10] revealed that YSi_2 was dimorphic, crystallizing in the hexagonal system (AlB_2 type) at low temperatures (β - YSi_2) and in the orthorhombic system (distorted form of the tetragonal $ThSi_2$ structure) at higher temperatures (α - YSi_2), which was reported that α - YSi_2 transformed to β - YSi_2 at 450°C [9]. However, the latest Y-Si phase diagram [1] shows the opposite designations for dimorphic YSi_2 , the low and high temperature phases are α - YSi_2 with the hexagonal crystal structure (space group: $P6/mmm$, AlB_2 type) and β - YSi_2 with the orthorhombic crystal structure (space group: $I4_1/amd$, distorted form of the tetragonal $ThSi_2$ type), respectively, and the phase transformation occurs at much higher temperature, 1242°C. This apparent contradiction would be due to the oxygen impurity derived from the raw

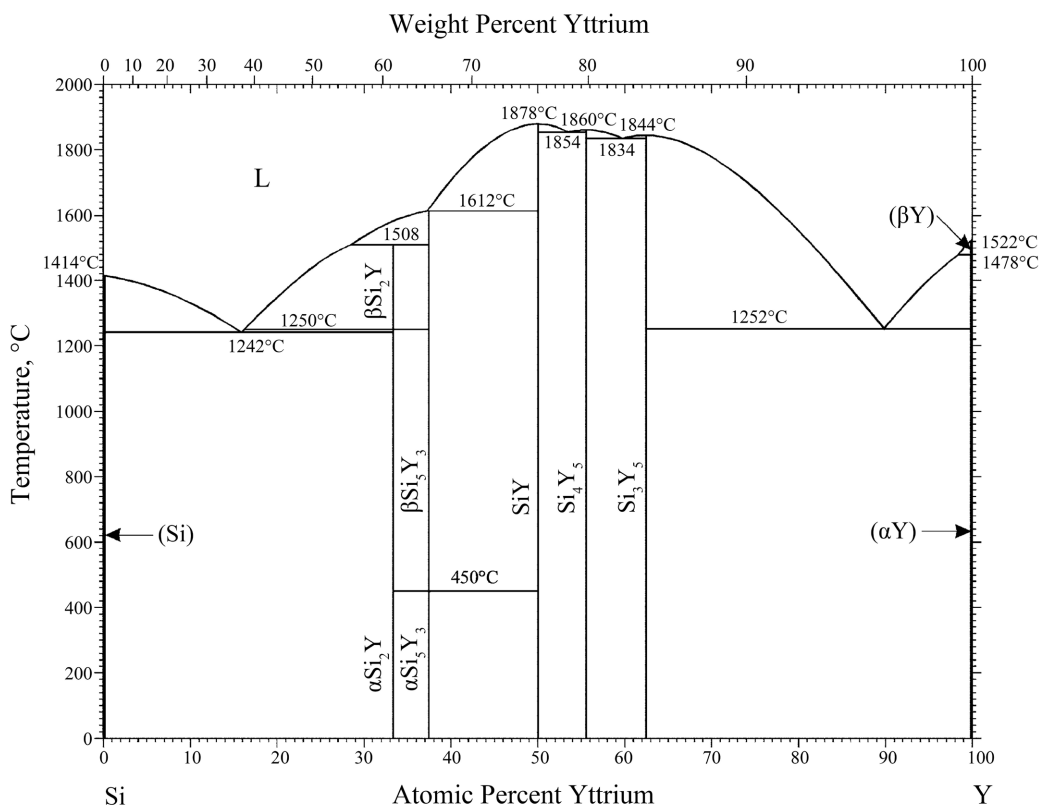


Figure 1. Y-Si phase diagram shown with a copy permission of Ref. [1].

metal powders. Even though the stoichiometric mixture of Y:Si = 1:2, was used for preparing the compound, partial oxidation of raw metal powders, especially that of Yttrium metal with much higher chemical activity, would make Yttrium metal to the mixture of Y and Y_2O_3 . Also, high purity Si metals, even if the semi-conductor grades were used, always contain oxygen impurity, since only metal impurities would be accounted for those applications. Thus, the vaporization of SiO at high temperature according to the chemical reaction (1) and/or during the silicothermic reduction of Y_2O_3 (2) would reduce the amount of Si [8].



These reactions would result in the formation of Y_3Si_5 instead of YSi_2 from the stoichiometric mixture of Y:Si = 1:2. Actually, the low temperature phase of Y_3Si_5 is the α - Y_3Si_5 with the orthorhombic crystal structure (space group: $I4_1/amd$, distorted form of the tetragonal $ThSi_2$ type), which is identical to that of the low temperatures form of β - YSi_2 reported in the early works [8] [9] [10]. This speculation would be further supported by the fact that the phase transformation from α - to β - Y_3Si_5 occurred at about 450°C, which was close to that of β - to α - YSi_2 reported in the earlier works [8] [9] [10]. It was reported that equiatomic phase YSi appeared to be little affected by oxygen, however, the presence of oxygen in high temperature preparations involving Y_3Si_5 could stabilize the hexagonal structure towards greater silicon deficiency and a Y:Si composition of Y_2Si_3 , whilst low temperature material of “ $Y_2Si_3O_x$ ” composition had an unstable orthorhombic structure [4]. Thus, the presence of oxygen during preparing the Y-Si compounds could significantly influence the phase identification results of this system.

It is well known that transition metal silicides possess good potential for use as structural materials at high temperature. However, high temperature oxidation behaviors about active metal silicides such as Yttrium Silicides have been rarely known. It was reported that undoped Y_5Si_3 showed poor oxidation resistance above 700°C due to mechanical failure under stresses produced by growth of the oxidation product [11]. Because YSi_2 nanoparticles incorporated SiGe exhibited excellent thermoelectric (TE) properties, the bulk YSi_2 was fabricated and the TE properties were investigated recently [12]. However, no physical and/or chemical properties have been reported for YSi_2 powder. The purpose of this work is to investigate the physical and chemical properties of YSi_2 powders fabricated by various methods paying special attention to their oxygen impurities.

2. Experimental

2.1. Synthesis of Low Purity YSi_2 Powders

Yttrium metal powder (purity > 99.9%, under 20 mesh; RARE METALLIC Co.,

Ltd.) supplied as paraffin slurry was separated by vacuum filtration and washed several times with hexane. Silicon powder (purity > 98.0%, under 100 mesh; Oxygen content = 1.03 wt%; Fuji film Wako Pure Chemical Corp.) was pulverized by using a rotary ball mill at 118 rpm for 8 hrs using 5 mm ϕ Si₃N₄ grinding media to give the Si powder having mean particle size of 8.3 μ m and oxygen content = 1.17 wt%. These powders were mixed with a composition of Y:Si = 1:2 in molar ratios, and uniaxially pressed at 30 MPa in a 15 mm diameter stainless-steel die. The powder compact was placed in a quartz tube (inner diameter = 10 mm and length = 10 cm), which was evacuated using a rotary pump, and then was sealed using a H₂-O₂ gas burner. The quartz tube was heat treated at 1100°C for 5 hr using a muffle furnace (Model MPC10; ADVANTEC Co., Ltd.) using the heating rate of 10°C/min.

The same powder compact was placed in a high purity BN crucible (N-1 grade; DENKA Company Ltd.), which was heat treated in a graphite susceptor at 1090°C for 5 hrs or 1500°C for 1 hr using a vacuum furnace with a graphite heating element (Model FVPHP-R-3; FUJIDENPA KOGYO Co., Ltd.). Heating and cooling rates were both 10°C/min.

2.2. Synthesis of High Purity YSi₂ Powders

A Yttrium metal ingot (purity > 99.9%, 7 - 10 mm; RARE METALLIC Co., Ltd.) was surrounded by Si powder (Purity > 99.99%, Oxygen content = 0.081 wt%, 300 μ m pass; KOJUNDO CHEMICAL LABORATORY Co., Ltd.) using the molar ratio of Y:Si = 1:2 in a high purity BN crucible (N-1 grade; DENKA Company Ltd.), which was heat treated in a graphite susceptor at 1600°C for 30 min under Ar atmosphere of 0.1 MPa using a furnace with a graphite heating element. Heating and cooling rates were both 10°C/min.

2.3. Chemical Stability Tests of YSi₂ Powders

A small amount of pulverized YSi₂ powder was immersed to various solvents including purified water, methanol (>99.8%), ethanol (>99.5%), propanol (>99.7%), butanol (>99.5%), benzene (>99.5%), *n*-hexane (>96.0%), or acetonitrile (>99.5%) in a test tube, and was settled for 24 hrs at room temperature under the atmospheric condition observing every hour whether the formation of bubbles or white precipitates occurred or not.

Low purity YSi₂ powder synthesized at 1500°C for 1 hr and high purity YSi₂ powder synthesized at 1600°C for 30 min were ground for 12 hrs by using a rotary ball mill at 120 rpm in various solvents including 2-propanol, *n*-hexane, or acetonitrile using 5 mm ϕ Si₃N₄ grinding media at room temperature under the atmospheric condition. After milling, each solvent was removed using a rotary evaporator, dried in an oven at 120°C for 6 hrs, and sieved by passing through a 60 mesh nylon sieve. The oxygen content of each powder was determined by the hot-gas extraction method (EMGA-920, HORIBA Ltd.) using 20 mg powder sample encapsulated with 1.0 g pure iron in a Ni crucible.

3. Results and Discussion

3.1. Fabrication Conditions and XRD Identification Results of Y-Si Compounds

Table 1 summarizes the fabrication conditions of Y-Si compounds with Y:Si = 1:2 composition indicating five powder samples as No. 1-5. **Figure 2** shows the XRD patterns of powder samples No. 1-4. Since the XRD pattern of sample No. 5 was found to be identical to that of No. 4, **Figure 2** lacks its XRD pattern. **Table 2** summarizes the XRD identification results of Y-Si compounds prepared in this work. **Table 3** and **Table 4** compare powder XRD peak data obtained in this work having 1) hexagonal and 2) orthorhombic, respectively, crystal structures with those of Powder Diffraction File (PDF) database registered by the International Centre for Diffraction Data (ICDD). As shown in **Table 2**, Y-Si compounds with hexagonal structure were identified as α -YSi₂ or β -Y₃Si₅, because both phases are indistinguishable with their XRD patterns as shown in **Table 3**.

It was reported that α -YSi₂ and β -Y₃Si₅ had the identical crystal structure, P6/mmm, and β -Y₃Si₅ had defect structure: Si atoms occupy 83% of the 2(d) positions in the space group P6/mmm [13]. Also, the orthorhombic phase was identified as β -YSi₂, because it was reported that the low temperature phase α -Y₃Si₅ (Ortho.) was stable below 450 °C [9]. Apparently, only sample No.2 has the orthorhombic structure, and the other samples possess the hexagonal structure as shown in **Table 3** and **Table 4**. Samples No. 1 and 2 were fabricated with low purity metal powders at virtually the same temperature around 1100 °C, at which the low temperature phase, α -YSi₂, should be stable according to the latest phase diagram [1]. However, sample No. 2 fabricated at 1090 °C in a graphite furnace under vacuum condition (continuously evacuated using a rotary pump) possesses the orthorhombic structure, the high temperature phase stable above 1250 °C. It would be noteworthy that sample No. 3 that was fabricated at 1500 °C in the same conditions other than temperature as sample No. 2 possesses the hexagonal structure, the low temperature phase stable below 1250 °C. This apparent contradiction would be explained as follows; Samples No. 1-3 were fabricated using Yttrium powder, which should contain a significant amount of surface oxygen, although the oxygen content was not accounted for its purity, 99.9% that accounted only for the total metal impurity. Because sample No.1 was fabricated in a sealed quartz tube (closed system), the vaporization of SiO according to Equation (1) would be suppressed. This speculation would be supported by the fact that weak peaks of Y₂O₃ are observed only in the sample No.1, which also shows middle intensities of Si peaks suggesting that Equation (1) be suppressed. However, when heat-treated under vacuum (open system), the amount of Si and Y₂O₃ would be reduced according to Equations (1) and (2), respectively, resulting the formation of unstable phase with orthorhombic structure [4] as observed for sample No. 2. When heat-treated at higher temperature, 1500 °C, this unstable Y₂Si₃O_x phase would decompose to the stable phases, β -Y₃Si₅ and Y₂SiO₅ and/or Y₂Si₂O₇ as shown in **Table 2**.

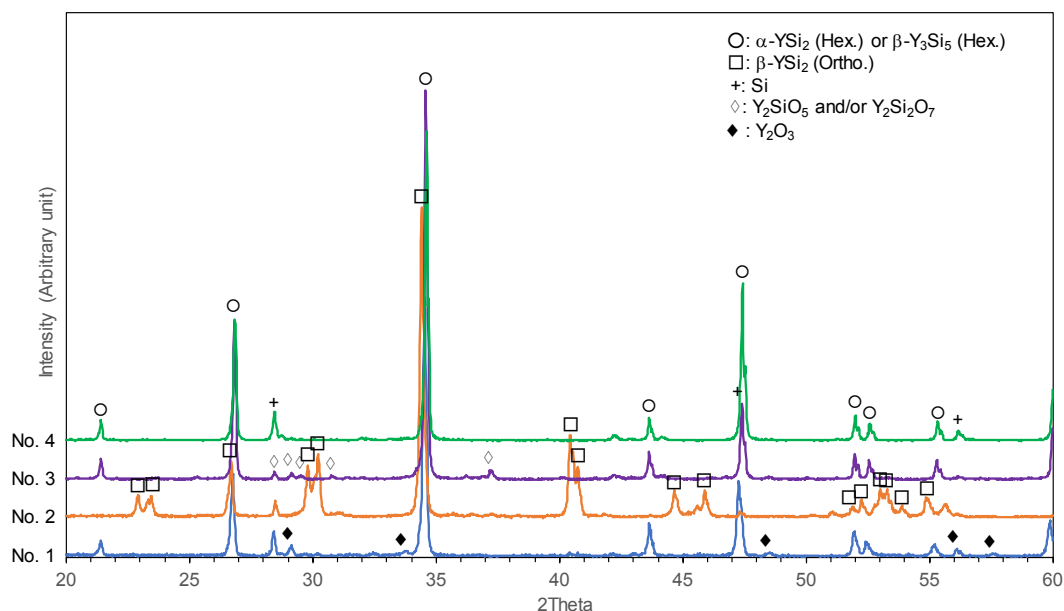


Figure 2. XRD patterns of Y-Si compounds fabricated in this work, sample No. 1-4, as shown in **Table 1**.

Table 1. Fabrication conditions of Y-Si compounds.

No.	Raw materials		Heat treatment conditions		
	Yttrium (purity)	Silicon [O]	Temp. (°C)	Duration hr	Atmosphere
1	powder (99.9%)	8.3 mm (1.17%)	1100	5	Quartz tube
2	powder (99.9%)	8.3 mm (1.17%)	1090	5	Vacuum
3	powder (99.9%)	8.3 mm (1.17%)	1500	1	Vacuum
4	ingot (99.9%)	-100 Me' (0.081%)	1600	0.5	0.1 MPa Ar
5	ingot (99.9%)	-100 Me' (0.081%)	1600 1100	0.5 12	0.1 MPa Ar

Table 2. XRD identification results of Y-Si compounds prepared in this work.

No.	XRD identification results					
	α -YSi ₂ (Hex.) ^{*1} or β -Y ₃ Si ₅ (Hex.) ^{*2}	β -YSi ₂ (Ortho.) ^{*1}	Si	Y ₂ SiO ₅ and/or Y ₂ Si ₂ O ₇	Y ₂ O ₃	
1	vs	—	m	tr	w	
2	—	vs	w	tr	—	
3	vs	—	w	w	—	
4	vs	—	m	—	—	
5	vs	—	m	—	—	

Intensity: vs: very strong; w: weak; vw: very weak; tr: trace; —: not detected. *1: It was reported that α -YSi₂ (No. 11-0596 in the PDF-ICDD) and β -YSi₂ (No. 11-0324 in the PDF-ICDD) were the low and high temperature phases, respectively, and the phase transformation occurred at 1250 °C [3]. *2: Since it was reported that the low temperature phase α -Y₃Si₅ (Ortho.) was stable below 450 °C [9], the high temperature phase β -Y₃Si₅ (No. 72-2173 in the PDF-ICDD) would be the stable phase in the current experimental condition. It was also reported that α -YSi₂ and β -Y₃Si₅ had the identical crystal structure, P6/mmm, and β -Y₃Si₅ had defect structure: Si atoms occupy 83% of the 2(d) positions in the space group P6/mmm [13].

Table 3. Comparison of powder XRD peak data obtained in this work with Powder Diffraction File (PDF) database registered by ICDD* (1) Hexagonal crystal structure.

PDF database				Sample No. and their powder XRD peak data obtained in this work		
Card No.	Compound	Card No.	Compound	No. 1	No. 3	No. 4
11-0596	YSi ₂	72-2173	Y ₃ Si ₅			
<i>hkl</i>	<i>2θ</i>	<i>hkl</i>	<i>2θ</i>	<i>2θ</i>	<i>2θ</i>	<i>2θ</i>
001	21.446	001	21.446	21.42	21.42	21.40
100	26.831	100	26.772	26.76	26.82	26.84
101	34.604	101	34.557	34.54	34.58	34.62
002	43.692	002	43.694	43.64	43.64	43.64
110	47.331	110	47.280	47.26	47.40	47.42
102	52.068	102	51.986	51.96	51.98	52.00
111	52.551	111	52.470	52.42	52.56	52.58
200	55.258	200	55.165	55.22	55.30	55.34
201	59.938	201	59.869	59.90	59.98	60<

Table 4. Comparison of powder XRD peak data obtained in this work with Powder Diffraction File (PDF) database registered by ICDD* (2) Orthorhombic crystal structure.

PDF database		Powder XRD peak data of sample No. 2
Card No.	Compound	
11-0324	YSi ₂	
<i>hkl</i>	<i>2θ</i>	<i>2θ</i>
101	22.961	22.92
011	23.452	23.46
004	26.748	26.70
103	29.856	29.80
013	30.271	30.22
112	34.465	34.40
105	40.413	40.44
015	40.795	40.74
200	44.69	44.64
020	45.86	45.90
116	52.194	52.24
204	52.942	53.00
107	53.208	53.28
017	53.409	53.44
024	53.781	53.90
213	54.933	54.96

When high purity metal powders were used, only diffraction peaks attributed to the hexagonal phase and Si are observed for sample No. 4 and almost no other oxide phases exist. A fact that the stoichiometric mixture of Y:Si = 1:2 resulted in the formation of Si suggests that the hexagonal phase would not be α -YSi₂ but β -Y₃Si₅, which would be further supported by the fact that the sample No. 4 was heat-treated at as high as 1600°C, at which this mixture should form a homogeneous liquid phase according to the Y-Si phase diagram [1]. To confirm whether the mixture of β -Y₃Si₅ and Si would be the stable phases for the stoichiometric mixture of Y:Si = 1:2, sample No.4 was further heat-treated at 1100°C for 12 hrs, which was specified as sample No. 5 in Table 1. This equilibration process did not change the diffraction peaks observed for sample No. 4 at all, supporting the hypothesis above mentioned. A recent study regarding the fabrication of YSi₂ by the spark plasma sintering at 1273 K under Ar atmosphere using high purity metals also demonstrated that the main peaks of all samples agreed well with the YSi_{1.67} standard pattern (JCPDS 01-071-3919), representing the single phase of the AlB₂-type structure (P6/mmm) [12]. It would be interesting that the 2θ values of all diffraction peaks of the β -Y₃Si₅ hexagonal lattice shifted in the order of sample No. 1 > No. 3 > No. 4 as shown in Table 3, suggesting that their lattice constants would be enlarged with the amount of oxygen impurity.

As described in the introduction, although the latest Y-Si phase diagram shown in Figure 1 [1] demonstrated that the α - and β -YSi₂ phases were the stable phases for the stoichiometric composition of Y:Si = 1:2 adopting the experimental results clearly showing their existences [6] [7]. However, a detailed investigation of phase equilibria and crystal structures of the compounds taking the effects of impurity oxygen into account demonstrated that the formation of YSi₂ was not confirmed as a stable phase [4]. As a result, there have been several phase diagrams for the Y-Si system. The current experimental results suggest that the high temperature phase with the hexagonal structure, β -Y₃Si₅, would be the stable phase even for the stoichiometric composition of Y:Si = 1:2, and that the high temperature phase with the orthorhombic structure, β -YSi₂, would be the meta-stable phase under high oxygen impurity content. Further investigation would be necessary to clarify the phase diagram of Y-Si system.

3.2. Chemical Stabilities of Y-Si Compounds

Figure 3 and Figure 4 show the appearances of α type and β type YSi₂ powders (sample No. 2 and 3), respectively, immersed in purified water, methanol, ethanol, propanol, butanol, benzene, *n*-hexane, or acetonitrile (from the left end to the right end) settled for 0, 1, 2, 4, 8, 16 and 24 hrs at room temperature under the atmospheric condition. It was observed that the dispersibility increased in the order as follows, water < methanol < ethanol < 2-propanol > butanol ~ acetonitrile >> benzene ~ *n*-hexane, demonstrating that the best solvent is propanol for dispersing YSi₂ powders. There was almost no difference between α type and β type YSi₂ powders, but β type showed better dispersibility after long duration, which would be due to the difference of mean particle sizes of both powders, 17

μm and $10\ \mu\text{m}$, respectively. It should be noted that neither the formation of bubbles nor white precipitates was observed in all solvents even after 24 hrs for both powders, suggesting much superior chemical stability to solvents of YSi_2 powders than Yttrium metal and Alkaline Earth Silicides, for which the formation of bubbles and/or white precipitates were observed in all solvents immediately after dispersing these powders.

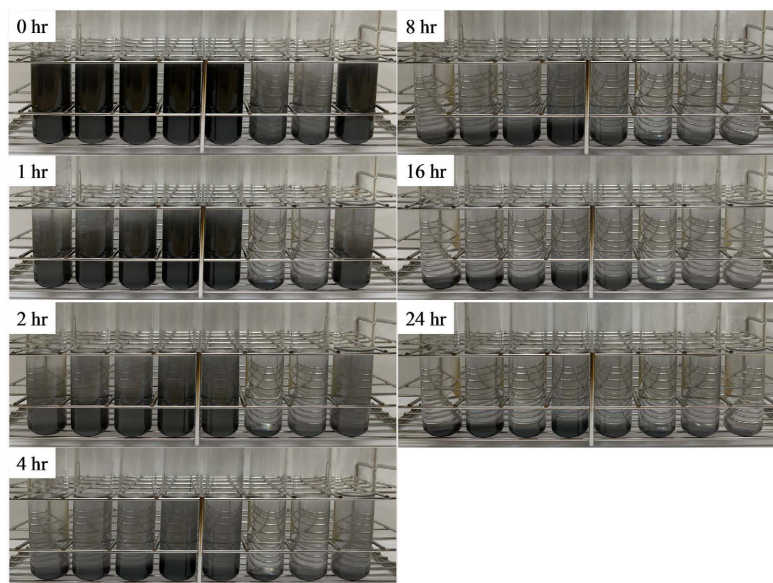


Figure 3. Appearances of α type powders (sample No. 2) immersed in purified water, methanol, ethanol, propanol, butanol, benzene, *n*-hexane, or acetonitrile (from the left end to the right end) settled for 0, 1, 2, 4, 8, 16 and 24 hrs at room temperature under the atmospheric condition.

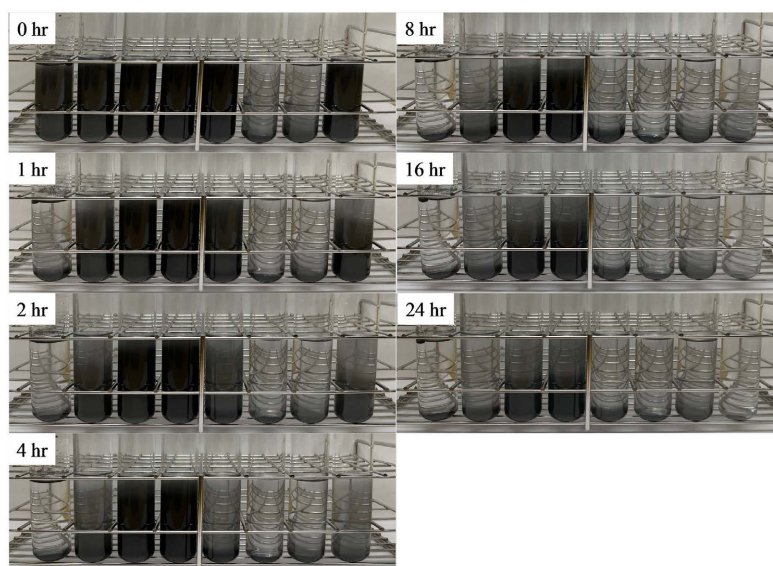


Figure 4. Appearances of β type powders (sample No. 3) immersed in purified water, methanol, ethanol, propanol, butanol, benzene, *n*-hexane, or acetonitrile (from the left end to the right end) settled for 0, 1, 2, 4, 8, 16 and 24 hrs at room temperature under the atmospheric condition.

Table 5. Oxygen contents of YSi₂ powders with low and high purity before and after grinding.

Powder sample	Oxygen content (wt%)			
	Before grinding	After grinding in		
	<150 μm	2-propanol	<i>n</i> -hexane	acetonitrile
No. 3 (low purity)	1.40	2.51	2.53	2.6
No. 4 (high purity)	0.59	1.70	1.39	1.77

Table 5 summarizes the oxygen contents of YSi₂ powders with low and high purity, sample No. 3 and 4 respectively, before and after grinding in 2-propanol, *n*-hexane or acetonitrile at room temperature under the atmospheric condition. It was confirmed that the use of high purity metals resulted in reducing oxygen content by about 58% comparing to the low purity metal powders. After grinding these powders in a ball mill at 120 rpm for 12 hrs using 2-propanol, *n*-hexane, or acetonitrile as solvents, the oxygen contents increased significantly probably due to the mechano-chemical effect. Although those solvents without hydroxy group, *n*-hexane and acetonitrile, were expected not to increase the oxygen content, there was almost no difference for oxygen contents after milling in all solvents for powder sample No. 3 (low purity). However, for powder sample No. 4 (high purity), *n*-hexane gave the least oxygen content probably due to its less contents of oxides such as Y₂SiO₅ and/or Y₂Si₂O₇ as shown in **Table 2**.

4. Conclusion

Y-Si compounds with the composition of Y:Si = 1:2 were fabricated using Yttrium and Silicon raw powders with low and high purity in various atmospheres and temperatures. Although the latest Y-Si phase diagram demonstrated that the α - and β -YSi₂ phases were the stable phases for the stoichiometric composition of Y:Si = 1:2, the current experimental results suggest that the high temperature phase with the hexagonal structure, β -Y₃Si₅, would be the stable phase even for the stoichiometric composition of Y:Si = 1:2, and that the high temperature phase with the orthorhombic structure, β -YSi₂, would be the meta-stable phase under high oxygen impurity content. It was demonstrated that YSi₂ powders possess much superior chemical stability than Yttrium metal. It was found that the best dispersing solvent for YSi₂ powder was 2-propanol.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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