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**DEVELOPMENT AND EVALUATION OF DIE AND CONTAINER
MATERIALS**

Fourth Quarterly Progress Report, July 1–September 30, 1978

By
R. R. Wills
D. E. Niesz

October 15, 1978

Work Performed Under Contract No. NAS-7-100-954876

Battelle Columbus Laboratories
Columbus, Ohio

U.S. Department of Energy

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Solar Energy

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FOURTH QUARTERLY PROGRESS REPORT
(Covering the Period July 1, 1978 to September 30, 1978)

on

DEVELOPMENT AND EVALUATION OF
DIE AND CONTAINER MATERIALS

JPL Contract No. 954876
Silicon Sheet Task
Low Cost Silicon Solar Array Project

to

JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY

October 15, 1978

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The JPL Low-Cost Silicon Solar Array Project is funded by DOE and forms part of the DOE Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays.

by

R. R. Wills, Principal Investigator, and D. E. Niesz, Program Manager

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ABSTRACT

Mass spectrometric studies of molten silicon in contact with mullite and $x = 0.75 \beta'$ Sialon show that the vapor pressures of Si, SiO, Al, and Al_2O species are very similar. In both cases the oxygen solubility is extremely low, but the aluminum content of the silicon is higher for the mullite-silicon system (3 weight percent), compared with 5000 ppm for the $x = 0.75 \beta'$ Sialon. The nitrogen concentration (312 ppm) of the silicon is marginally below that for the Si_3N_4 -silicon system indicating that the addition of aluminum and oxygen to $\beta \text{Si}_3\text{N}_4$ does stabilize the nitrogen to some extent.

Silicon beryllium oxynitrides were fabricated by hot pressing Si_3N_4 -BeO-SiO₂ mixtures using three Si_3N_4 powders. Single phase materials were obtained with KBI, Inc. and Sylvania SN502 grade powders, but not with Sylvania SN402 grade powder.

Preliminary evaluation of the silicon beryllium oxynitride materials with molten silicon did not reveal any attack of these materials. $X = 0.75 \beta'$ Sialon did, however, undergo slight attack. It is suggested that the rate of dissolution of aluminum in molten silicon could be affected by whether the Sialon is used in an inert atmosphere or under vacuum conditions.

ACKNOWLEDGEMENT

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DEVELOPMENT AND EVALUATION OF DIE AND CONTAINER MATERIALS

by

R. R. Wills and D. E. Niesz

INTRODUCTION

The JPL Low Cost Silicon Solar Array Project has been established with the goal of decreasing the cost of solar photovoltaic arrays for electrical power generation. Methods of producing silicon sheet for solar cells are under active development as one of several tasks designed to achieve this objective. In the crystal-growing processes a refractory crucible is required to hold the molten silicon, while in the ribbon processes an additional refractory shaping die is needed to enable silicon ribbon to be produced. In several ribbon processes the high-temperature materials are a limiting factor in the development of the technique.

The objective of this study is to develop and evaluate refractory die and container materials. The performance targets for the die and container materials are given in the statement of work as:

- (a) The material must be mechanically stable to temperatures greater than the melting point of silicon (1412 C). Thus, it must not melt or undergo other destructive phase changes below this temperature.
- (b) Materials in contact with molten silicon must be dimensionally stable, to 0.5 mil over a 24-hour period in case of dies. This is necessary to maintain dimensional control of the processed silicon strip, and is to include erosion, corrosion, or growth of surface reaction products. With container materials, acceptable reaction rates will be controlled by permissible impurity level.

- (c) The die and container material must not excessively contaminate silicon processed through it. Present indications suggest that 10^{15} atoms/cm³ is an upper limit for general impurities. Exceptions to this are: aluminum, phosphorus, boron, arsenic, and gallium, which may be present 1 or 2 orders of magnitude higher, and carbon, oxygen, and nitrogen, which may be present in amounts dictated by erosion rates (approximately 10^{19} atoms/cm³). However, revision of these numbers may occur as knowledge of the specific effects of these elements is developed. For example, there are indications that structural imperfections result from carbon levels greater than 1×10^{18} atoms/cm³.
- (d) The process or processes developed must be amenable to the fabrication of dies and containers with close tolerances and of varying geometries.
- (e) The die to be produced and evaluated on this program shall be capable of producing and maintaining a capillary column of silicon 1 to 3 cm wide x 0.1 cm thick to a height of at least 2.5 cm. Experience with other materials has indicated that a contact angle of less than 80 degrees is required.

This study has been initiated to attempt to meet these requirements. The general approach involves the determination of the solution thermodynamics of several refractory materials in contact with molten silicon, together with the development and assessment of silicon metal oxynitride ceramics.

These silicon metal oxynitride ceramics are two silicon aluminum oxynitrides (Sialons) and one silicon beryllium oxynitride (Sibon). Each material is a solid solution, not a discrete compound. The compositions of these silicon metal oxynitride solid solutions are represented by the following formulae:

The first material, frequently called β' Sialon, is a solid solution between β Si_3N_4 and $\text{Al}_2\text{O}_3 \cdot \text{AlN}$. The second material referred to as O' Sialon is a solid solution between $\text{Si}_2\text{N}_2\text{O}$ and Al_2O_3 . The third material is a solid solution between β - Si_3N_4 and Be_2SiO_4 . This solid solution has been designated β' Sibeon by the authors.

These materials will be prepared by the following reactions:

In the initial phase of the program commercial high purity Si_3N_4 , Al_2O_3 , and SiO_2 powders were obtained and analyzed, and the preparation of AlN powder by a gas phase nucleation process was investigated. This process gave low yields of AlN powder and consequently commercial AlN powder was used in the preparation of β' Sialons.

Well controlled processing schedules were developed for the preparation of single phase β' and O' Sialons. These procedures were adopted after the importance of the nature of Si_3N_4 powder was discovered. The oxygen content, chlorine content, and particle size of the Si_3N_4 powder in each mixture were found to strongly affect reactivity and densification during hot pressing.

In contact with molten silicon O' Sialons formed an interface zone consisting of a Si_3N_4 layer and an amorphous layer of composition 90.7 percent silicon, 0.16 percent aluminum, and 9.14 percent oxygen and nitrogen. Thin aluminum rich precipitates also form in the silicon. The stability of β' Sialons in contact with molten silicon is dependent upon their aluminum content. β' Sialons of composition $x = 1$ appeared to be stable, but at higher aluminum contents thin precipitates formed in the silicon.

Mass spectrometric studies of molten silicon in contact with SiO_2 , BeO , Al_2O_3 , Si_3N_4 , and SiC were completed. Henry's Law Constants for oxygen, beryllium, aluminum, and nitrogen at concentrations ≤ 5000 ppm were determined and the solubilities of oxygen and nitrogen in molten silicon were found to be in good agreement with values given in the literature.

TECHNICAL DISCUSSION OF RESULTS THIS QUARTER

Solution Thermodynamics

Mass spectrometric studies of mullite* and $x = 0.75$ β' Sialon crucibles in contact with molten silicon have been completed this quarter. Table 1 shows the observed vapor pressures and other analytical data obtained from melting silicon in mullite and holding at 1700 K for 3 hours.

TABLE 1. DATA FROM Si - MULLITE REACTION
AT 1700 K FOR 3 HOURS

$P_{SiO} = 1.5 \times 10^{-6}$ atm
$P_{Si} = 8.3 \times 10^{-8}$
$P_{Al_2O} = 1.8 \times 10^{-8}$
$P_{Al} = 3.2 \times 10^{-9}$
$P_{O_2} = 5 \times 10^{-28}$
[O] in Si = 0.2 ppm
[Al] in Si = .3 percent

The oxygen concentration shown in Table 1 is a calculated value, because oxygen was not detected by the mass spectrometer. The calculation was carried out using Henry's Law Constant obtained for oxygen when silicon was melted in Al_2O_3 .^{(1)**} All vapor pressure values obtained from silicon in mullite were lower than either silicon in SiO_2 or silicon in Al_2O_3 . Table 2 shows the impurities in the silicon detected by spark source mass spectrographic analysis (SSMS).

* Fabricated from mullite supplied by Coors Porcelain Company. Material believed to contain approximately 30 percent glass phase.

** References are located on Page 18.

TABLE 2. THE IMPURITIES DETECTED BY SSMS IN SILICON

Impurities Detected from Si Melted in Mullite	
Na	800 ppm
Mg	10
Al	3 percent
P	20 ppm
Ti	200
Cr	5
Fe	1000
Ni	30
Cu	10

Table 3 shows the data obtained from silicon melted in a $x = 0.75 \beta'$ Sialon crucible. This system was held at 1700 K for 3 hours, as in previous experiments.

TABLE 3. DATA OBTAINED FROM SILICON MELTED IN A $x = 0.75 \beta'$ SIALON

P Si	5.5×10^{-8} atm
P SiO	7.0×10^{-7}
P N ₂	3.4×10^{-7}
P Al	3.7×10^{-9}
P Al ₂ O	4.3×10^{-8}
[O] in Si (Calc)	0.1 ppm
[N] in Si	312 ppm
[Al] in Si	0.5 percent

The oxygen concentration in the silicon melted in the β Sialon crucible was below the detection limit of the mass spectrometer. The value shown in Table 3 is calculated using Henry's Law Constant obtained for oxygen from silicon melted in Al_2O_3 . Table 4 shows the impurities in the silicon after melting in the β Sialon crucible.

TABLE 4. IMPURITIES IN SILICON AFTER
MELTING IN β SIALON CRUCIBLE

Impurities	
Al	5000 ppm
Na	500 ppm
Mg	170
S	70
Cl	50
K	100
Cr	50
Fe	100
Ni	300
Cu	70
Zn	100

Compared with the silicon- Si_3N_4 system the partial pressures of N_2 and $\text{Si}^{(2)}$ are almost identical, but the partial pressure of SiO is two orders of magnitude higher. The oxygen concentration of the silicon (0.1 ppm) is very low, and below the value for the as-received material (4 ppm). Interestingly the nitrogen concentration (312 ppm) is marginally below that for Si_3N_4 (360 ppm) indicating that the addition of Al and O to β Si_3N_4 does stabilize N to some extent. The amount of stabilization probably depends on the Al and O concentrations in the lattice.

Figure 1 shows some attack of the $x = 0.75$ β' Sialon, but the concentration of dissolved aluminum in silicon (5000 ppm) is approximately an order of magnitude higher than would be expected if a 10μ surface layer of Sialon dissolved in the silicon. A second value, also determined by spark

500X

6J580

FIGURE 1. SILICON - $X = 0.75 \beta'$ SIALON INTERFACE
SHOWING SOME ATTACK BY THE SILICON

source analysis, was even higher (2 weight percent). In view of the scatter in values of aluminum concentration, it is planned to check these values against the solubility of aluminum calculated from Henry's Law Constant after the activity of a suitable aluminum vapor species has been determined.

The vapor pressure data suggest that the loss of SiO from β' Sialon leaves the surface aluminum rich, thus accelerating dissolution into the molten silicon. As this experiment was performed under vacuum, it is likely that SiO volatilization will be retarded under atmospheric pressure, thus diminishing the rate of aluminum dissolution. It could thus be quite critical whether a β' Sialon die is used under vacuum conditions or in an inert gas atmosphere.

Assuming the solubility of aluminum in molten silicon for the system silicon - x = 0.75 β' Sialon is correctly 5000 ppm, then the performance of this material is comparable to that of SiC (carbon solubility 7000 ppm) and BeO (beryllium solubility 5000 ppm). As discussed later, this x = 0.75 β' Sialon has also been held in contact with molten silicon under an argon atmosphere, but because of different reaction times and temperatures, meaningful comparison between the experiments is not possible.

The above experiments with x = 0.75 β' Sialon indicate that it is a possible die material, but its use may depend upon the rate of flow of silicon across the die and whether the ribbon growing process is performed under vacuum or in an inert atmosphere.

Fabrication of β' Sibeons (β' Silicon Beryllium Oxynitrides)

β' Sibeon is a solid solution of Be_2SiO_4 in $\beta\text{-Si}_3\text{N}_4$. Since Si_3N_4 powder contains a small amount of SiO_2 the lower practical solubility limit of beryllium in β' Sibeon is determined by this SiO_2 concentration unless the SiO_2 is removed. For compositions above this lower level SiO_2 must be added to the Si_3N_4 (SiO_2) + BeO mixture so that the total SiO_2 content gives a SiO_2/BeO molar ratio of 0.5. The maximum solubility of Be_2SiO_4 in $\beta\text{-Si}_3\text{N}_4$ at 1760 C is 19 mole percent. ⁽³⁾

Three Si_3N_4 powders were used to prepare β' Sibeons of different beryllium contents. Two of the powders, GTE Sylvania's SN402 grade and SN502, have been described ^(3,4) in studies of the preparation of α' and β' Sialons.

The third powder, Kawecki Berylco's high purity grade Si_3N_4 powder, was selected because its low oxygen concentration (0.79 weight percent) would enable a very dilute β' Sibeon solid solution to be formed. The concentration of metallic impurities (see Table 5) in this powder is similar to that in Sylvania's SN502 grade powder except for higher concentrations of aluminum (200 ppm) and iron (400 ppm). The BeO powder, UOX grade supplied by Brush Wellman, Inc., had an average particle size of 1.5 μm , and the SiO_2 used in the preparation of one sample was Cabosil EH-5 grade SiO_2 .

Fifty to seventy-five g powder batches were prepared by ball milling for 2-4 hours in polyethylene jars using Al_2O_3 balls and n-hexane media. After vacuum drying, each batch was stored under nitrogen in sealed containers.

Table 6 lists the β' Sibeon samples prepared to date. A simple hot pressing cycle was used with the SN502 and KBI Si_3N_4 powders, and the modified hot pressing cycle⁽²⁾ was employed with the batch containing SN402 Si_3N_4 powder. This latter sample was not a single phase material, probably because chlorine in the Si_3N_4 powder reacted with the BeO to form the volatile compound BeCl_2 . The second phase has been designated $0'$ but it is not known whether the phase is a solid solution of beryllium silicate in silicon oxynitride or silicon oxynitride itself.

Specimens prepared from SN502 Si_3N_4 and BeO appeared to contain a considerable number of agglomerates as though the powders had not mixed well during ball milling. Adding a small amount of Cabosil SiO_2 resulted in better mixing, aided densification, and resulted in a homogeneous sample. Spark source analysis of these materials has not yet been completed.

Fabrication of β' Sialons (β' Silicon Aluminum Oxynitrides)

In the last Quarterly Report a well controlled process for fabricating single phase β' Sialons was described. The key feature of this process is the removal of a chlorine-nitrogen containing species at 1500 C and the subsequent nitridation of free silicon in the powder compact before hot pressing. In an effort to learn more about the nature of the volatile species a Si_3N_4 - Al_2O_3 -AlN mixture corresponding to $x = 1.0$ β' Sialon was examined in the Knudsen cell, using a BN crucible to hold the powder sample.

The results in Table 7 show that interaction between the crucible and the vapor species has occurred, making data interpretation a difficult task. The results, however, do show that hydrogen is present in the mixture, probably in the Si_3N_4 powder itself.

TABLE 5. MASS SPECTROGRAPHIC ANALYSIS OF KBI
HIGH PURITY GRADE POWDER

Element	KBI Lot 523-87-2	Element	KBI Lot 523-87-2
Li	0.05	Ag	<0.1
Be	0.03	Cd	<0.1
B	7	In	<0.02
F	1	Sn	0.7
Na	5	Sb	<0.02
Mg	<10	Te	<0.1
Al	200	I	<0.1
Si	Major	Cs	<0.05
P	5	Ba	2
S	6	La	<0.03
Cl	10	Ce	<0.2
K	1	Pr	<0.02
Ca	<30	Nd	<0.1
Sc	<0.2	Sm	<0.1
Ti	20	Eu	<0.04
V	0.2	Gd	<0.05
Cr	30	Tb	<0.01
Mn	7	Dy	<0.1
Fe	400	Ho	<0.02
Co	<0.2	Er	<0.07
Ni	20	Tm	<0.1
Cu	7	Yb	<0.2
Zn	<0.2	Lu	<0.03
Ga	4	Hf	<0.1
Ge	<0.3	Ta	<0.1
As	0.05	W	<0.1
Se	<0.1	Re	<0.1
Br	<0.03	Os	<0.1
Rb	0.1	Ir	<0.1
Sr	0.2	Pt	<0.1
Y	0.1	Au	<0.04
Zr	0.1	Hg	<0.1
Nb	0.2	Tl	<0.2
Mo	1	Pb	<0.03
Ru	<0.1	Bi	<0.02
Rh	<0.1	Th	<0.02
Pd	<0.3	U	<0.02

TABLE 6. COMPOSITIONS, DENSITIES, AND PHASE ANALYSIS OF HOT
PRESSED SILICON BERYLLIUM OXYNITRIDE SAMPLES

Starting Mixture	Concentration of Be_2SiO_4 (mol percent)	Density (g/cm^3)	Phase Analysis
KBI $\text{Si}_3\text{N}_4 + \text{BeO}$	3.447	3.0	β'
SN502 $\text{Si}_3\text{N}_4 + \text{BeO}$	5.863	3.02	β'
SN502 $\text{Si}_3\text{N}_4 + \text{BeO} + \text{SiO}_2$	7.522	3.17	β'
SN402 $\text{Si}_3\text{N}_4 - \text{BeO}$	11.948	3.11	$\beta' + \alpha'$

Note: Assuming the density of β' Sibeon is $3.18 \text{ g}/\text{cm}^3$, all the
above samples have densities >94 percent theoretical.

TABLE 7. VAPOR SPECIES ABOVE $\text{Si}_3\text{N}_4\text{-Al}_2\text{O}_3\text{-AlN}$ MIXTURE AS A FUNCTION OF TEMPERATURE

Temperature	Species	Pressure
1075 C	SiO	1.5×10^{-9} atm
	HCl	3×10^{-10}
1190	SiO	9×10^{-9}
	HCl	1×10^{-10}
1330	SiO	8×10^{-8}
	HCl	1×10^{-10}
	BOCl	5×10^{-9}
	BOCl_2	9×10^{-10}
	N_2	4×10^{-8}
1420	SiO	1×10^{-7}
	HCl	2×10^{-9}
	BOCl	6×10^{-7}
	B_2Cl_2	5×10^{-10}
	N_2	6×10^{-8}
1550	N_2	1×10^{-7}
	SiO	1×10^{-7}
	BOCl	1×10^{-7}
	BOCl_2	4×10^{-9}
	HCl	1×10^{-9}
	Al	4×10^{-7}
	Al_2O	1×10^{-7}
1770	B_2Cl_2	1×10^{-9}
	SiO	9×10^{-8}
	N_2	2×10^{-6}
	Al	4×10^{-7}
	BOCl	4×10^{-11}
	Al_2O	8×10^{-8}

$x = 0.75 \beta'$ Sialon is considered to be the lower composition limit for practical fabrication purposes due to difficulties in obtaining high density single phase material with decreasing Al_2O_3 content and also compensating for the SiO_2 content of the Si_3N_4 powder in the powder mixture. Fabrication of $x = 0.75 \beta'$ Sialon has been scaled up from 1 inch diameter discs to 2-5/8 inch diameter discs.

Compatibility With Molten Silicon

β' Sialon

$x = 0.75 \beta'$ Sialon, prepared from SN402 Si_3N_4 powder and Al_2O_3 and AlN powders using the modified hot pressing procedure, was held in contact with molten silicon at 1450 C for one hour under an argon atmosphere. Figures 2 and 3 show the interface region between the silicon and the ceramic. While there is no evidence of precipitates in the silicon, some attack of the Sialon has occurred (see Figure 3). The degree of attack is less than observed in the Knudsen cell experiment (see Figure 1), but the exact reason for this is not known at this time since the experiments were performed under different temperature-time-atmosphere conditions.

β' Sibeons

All four β' Sibeons described in Table 6 were exposed to molten silicon at 1450 C for one hour under an argon atmosphere. Preliminary analysis by optical metallography at low magnification does not show any attack of the ceramic (see Figures 4-7). This is also true for the two phase material ($\beta' + 0'$), as shown in Figure 5. While more detailed evaluation of β' Sibeon materials has yet to be done, this initial examination does indicate their potential as a die material.

100X

6J252

FIGURE 2. INTERFACE BETWEEN $X = 0.75 \beta'$ SIALON AND SILICON
AFTER ONE HOUR AT 1450 C UNDER ARGON ATMOSPHERE

100X

6J253

FIGURE 3. ATTACK OF $X = 0.75 \beta'$ SIALON BY MOLTEN SILICON
AFTER ONE HOUR AT 1450 C UNDER ARGON ATMOSPHERE

100X

6J654

FIGURE 4. INTERFACE BETWEEN β' SIBEON SAMPLE, FABRICATED FROM SN502 Si_3N_4 , BeO AND SiO_2 , AND SILICON AFTER ONE HOUR AT 1450 C UNDER ARGON ATMOSPHERE

100X

6J244

FIGURE 5. INTERFACE BETWEEN β' SIBEON SAMPLE, FABRICATED FROM SN402 Si_3N_4 + BeO, AND SILICON AFTER ONE HOUR AT 1450 C UNDER ARGON ATMOSPHERE

100X

6J655

FIGURE 6. INTERFACE BETWEEN β' SIBEON, FABRICATED FROM KBI, INC. $\text{Si}_3\text{N}_4 + \text{BeO}$, AND SILICON AFTER ONE HOUR AT 1450 C UNDER ARGON ATMOSPHERE

100X

6J658

FIGURE 7. INTERFACE BETWEEN β' SIBEON, FABRICATED FROM SN502 $\text{Si}_3\text{N}_4 + \text{BeO}$, AND SILICON AFTER ONE HOUR AT 1450 C UNDER ARGON ATMOSPHERE

WORK NEXT PERIOD

- (1) Mass spectrometric studies of the interaction of molten silicon with β' Sibeon.
- (2) Perform capillary rise test with $x = 0.75 \beta'$ Sialon.
- (3) Fabricate $x = 0.75 \beta'$ die.
- (4) Complete processing studies on the fabrication of β' Sibeon.
- (5) Select optimum β' Sibeon composition, and scale up hot pressings to 2-5/8 inch diameter sample size. Perform capillary rise test.
- (6) Fabricate β' Sibeon die.

NEW TECHNOLOGY

No items of new technology have been specifically reported so far.

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