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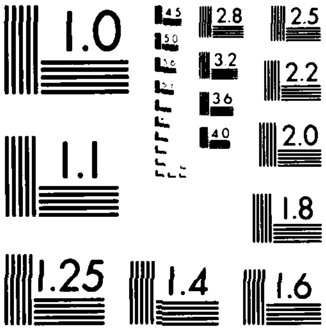

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TECHNICAL REPORT No. 11

THIN FILMS PROPERTIES OF SPUTTERED NIOBIUM SILICIDE ON SiO_2, Si_3N_4 and on N+ Poly-Si

by

T. P. Chow*, W.-J. Lu, A. J. Steckl, B. J. Baliga*

Prepared for Publication
in the
Journal of the Electrochemical Society

Rensselaer Polytechnic Institute
Center for Integrated Electronics
Troy, NY 12181

March 1985

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The thin film properties of sputtered niobium silicide on SiO_2, Si_3N_4 and $n^{(+)}$ doped poly-Si have been investigated. The structural and compositional properties were studied with X-ray diffraction, Rutherford backscattering spectrometry and secondary ion mass spectrometry.

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THIN FILM PROPERTIES OF SPUTTERED
NIOBIUM SILICIDE
ON SiO_2, Si_3N_4 AND ON N^+ POLY-SI

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ABSTRACT

The thin film properties of sputtered niobium silicide on SiO_2, Si_3N_4 and n^+ doped poly-Si have been investigated. The structural and compositional properties were studied with X-ray diffraction, Rutherford backscattering spectrometry and secondary ion mass spectrometry.

I. INTRODUCTION

Refractory metal silicides have become a key part of VLSI silicon device technology as MOS gates and interconnects due to their low resistivity, oxidation resistance and compatibility with MOS processes [1,2]. Among the silicides, the most widely used ones are $MoSi_2$, $TaSi_2$, WSi_2 and $TiSi_2$. Recently, $NbSi_2$ was also reported [3] to have similar properties as the other refractory silicides. In that study, slightly metal-rich (Si/Nb ~1.8) silicide films obtained from a hot-pressed composite target in rf sputtering system were investigated. Those films were shown to contain $NbSi_2$ as well as a significant amount of hexagonal Nb_5Si_3. Also, a resistivity of ~ 100 μΩ-cm was measured after annealing at elevated temperatures.

In this paper, we report on improved thin film properties of niobium silicide on SiO_2 and on n^+ poly-Si.

II. EXPERIMENTAL PROCEDURE

Silicon-rich (Si/Nb ~ 2.3) silicide films were deposited at room temperature from a cold-pressed composite target (99.6%) in a dc magnetron sputtering system (Varian 3140). Rutherford Backscattering Spectrometry (RBS) was done with a 2 MeV $^4He^+$ beam from a linear accelerator, Secondary Ion Mass Spectrometry with a Cameca IMS 3-f ion microscope and X-ray diffraction with a Siemens D-500 automated diffractometer.

III. RESULTS

(a) Structure and Composition -

The as-deposited films were essentially amorphous but after annealing at elevated temperatures, they exhibited a predominantly $NbSi_2$ structure. Fig. 1a and b show X-ray diffraction patterns for an as-sputtered film and the same film after annealing in hydrogen at 1000°C for 60 min. The major niobium disilicide peaks are at 2θ of 21.4, 25.4, 40.1, 41.2 and 47.0°, corresponding to the (100), (101), (111), (003) and (112) planes repectively. Trace amounts of carbon-stabilized, hexagonal Nb_5Si_3 components were also detected at 27.2, 29.2 and 36.4°. Pure Nb_5Si_3 is stable in two tetragonal structures ($D8_1$ (tI32) and $D8_m$ (tI32)) while impurities, such as carbon, tends to stabilize it into the hexagonal structure ($D8_8$) [4]. In fact, many of the other metal silicides (Mo_5Si_3, W_5Si_3, and others) have the same characteristic and the detection of such a hexagonal silicide phase is indicative of the presence of carbon or other impurities.

(b) Resistivity -

The thin film resistivity of these $NbSi_2$ films after annealing was observed to be consistently lower than the previously reported films. In Fig. 2, the sheet resistance of 5600Å niobium silicide on oxidized silicon substrates is shown as a function of annealing time at 800, 900 and 1000°C in hydrogen. As deposited, the films have a R_s of ~11 Ω/square. The resistivity decreases rapidly within the first 15 min and changes only slightly for longer times. After annealing for 1 hr, the sheet resistance dropped to 2.8, 2.0 and 1.3 Ω/square for 800, 900 and 1000°C respectively. For the silicide film on SiO_2, the sheet resistance dropped from 10.5 Ω/square to 1.4 Ω/square (78 μΩ-cm) after 15 min and to 1.3 Ω/square (72 μΩ-cm) after 60 min. The lowest $NbSi_2$ resistivity measured after annealing at 1000°C was ~ 70 μΩ-cm which represents a ~ 30% improvement over previous data on rf sputtered films and is close to the value of ~ 50 μΩ-cm reported for niobium on poly-Si films annealed under vacuum [1]. The bulk values, for comparison, range from 6.3 to 50 μΩ-cm, with the lower values probably erroneous [4]. Similar dependence on annealing time has been observed for niobium silicide on silicon nitride and on n^+ poly-Si (polycide) and is shown in Fig. 3 and 4. For the polycide stack, the sheet resistance was decreased from 13.7 to 2.8 Ω/square after 30 min but increased to 3.0 Ω/square after 60 min. The rapid decrease in resistivity followed by a saturation-like characteristic when annealed at high temperatures (>700°C) has also been observed for other silicides, such as $TiSi_2$ [5,6], $MoSi_2$ [7,8] and WSi_2 [8,9,10]. Also, when these niobium silicide films were annealed at different temperatures, similar dependence of sheet resistace on time was observed but higher saturation values were obtained for lower temperatures.

approx. microohms

IV. SUMMARY

Niobium silicide thin films deposited on SiO_2, Si_3N_4 and $n^{(+)}$ poly-Si have been characterized. Similar to the other refractory silicides, annealing at high temperatures resulted in structural recrystallization and a sharp decrease in resistivity. After annealing at 1000°C, a resistivity of ~70μΩ-cm was obtained. For a 2500Å $NbSi_2$/2500Å N^+/poly-Si stack, a sheet resistance of 2.5 Ω/square after annealing at the same temperature.

AKNOWLEDGMENT

One of us (AJS) would like to acknowledge the Office of Naval Research for its partial support of this work.

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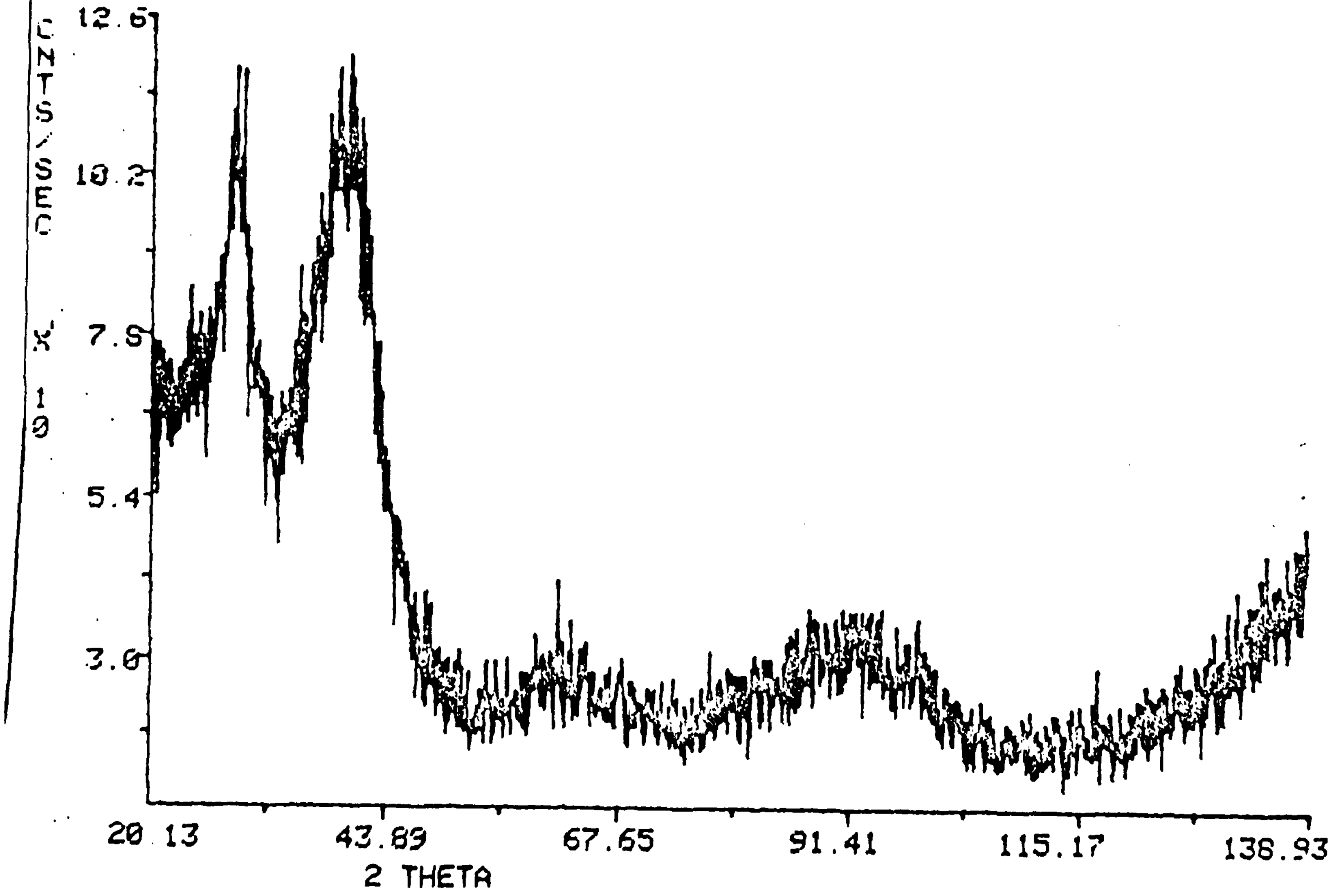
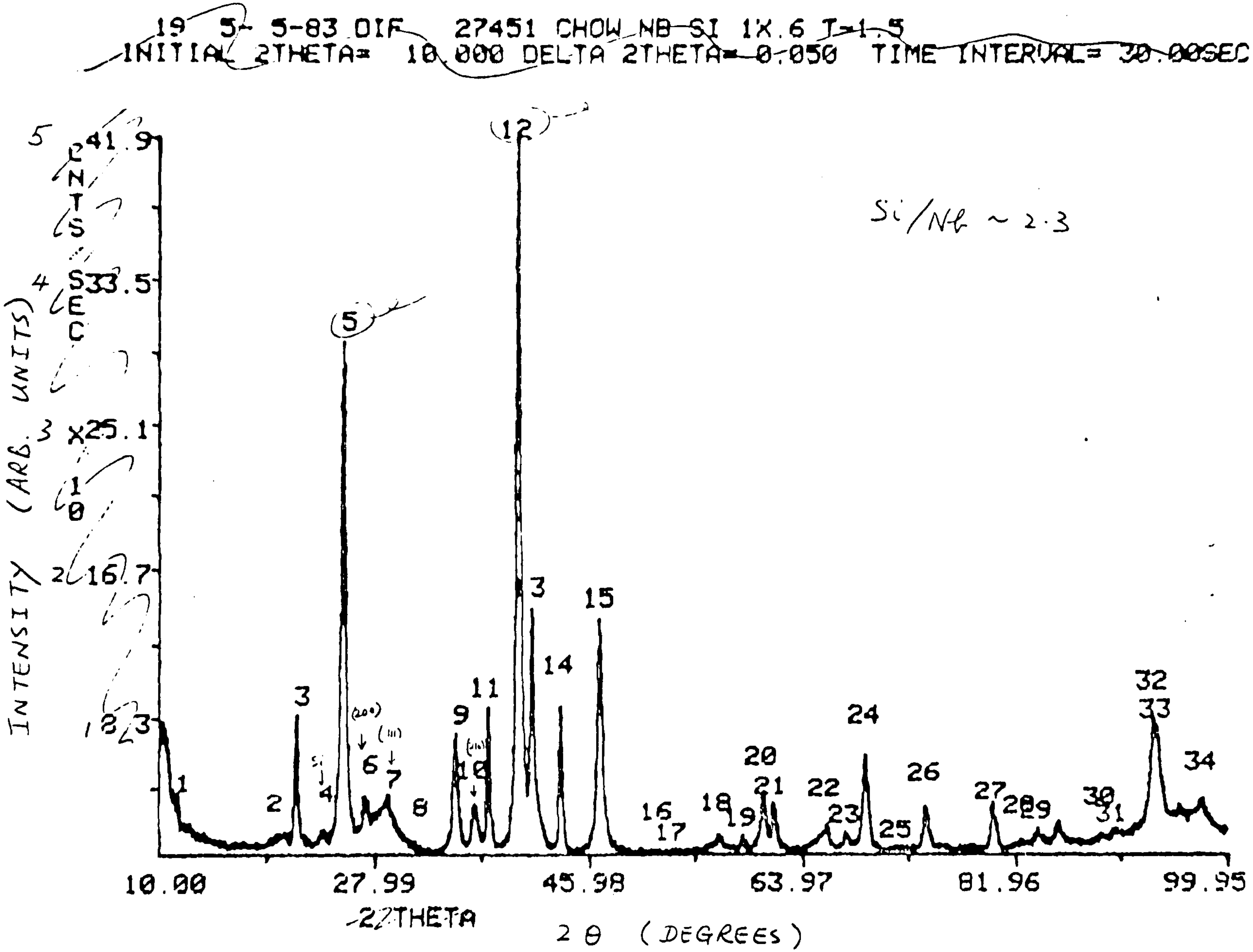
LIST OF FIGURES AND TABLES

7 10- 4-83 DIF 27657 PAUL CHOW 1X.6 50/20 T/2T
INITIAL 2THETA= 20.000 DELTA 2THETA= 0.100 TIME INTERVAL= 2.00SEC




19 5- 5-83 DIF 27451 CHOW NB-SI 1X.6 T-1.5
INITIAL 2THETA= 10.000 DELTA 2THETA= 0.050 TIME INTERVAL= 30.00SEC
CNTS SEC X 10
41.9
33.5
25.1
16.7
8.3
5
4
3
2
1
INTENSITY (ARB. UNITS)
10.00
27.99
45.98
63.97
81.96
99.95
2THETA
2 θ (DEGREES)
1
2
3
Si
4
5
(200)
6
(111)
7
8
9
(210)
10
11
12
3
14
15
16
17
18
19
20
21
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23
24
25
26
27
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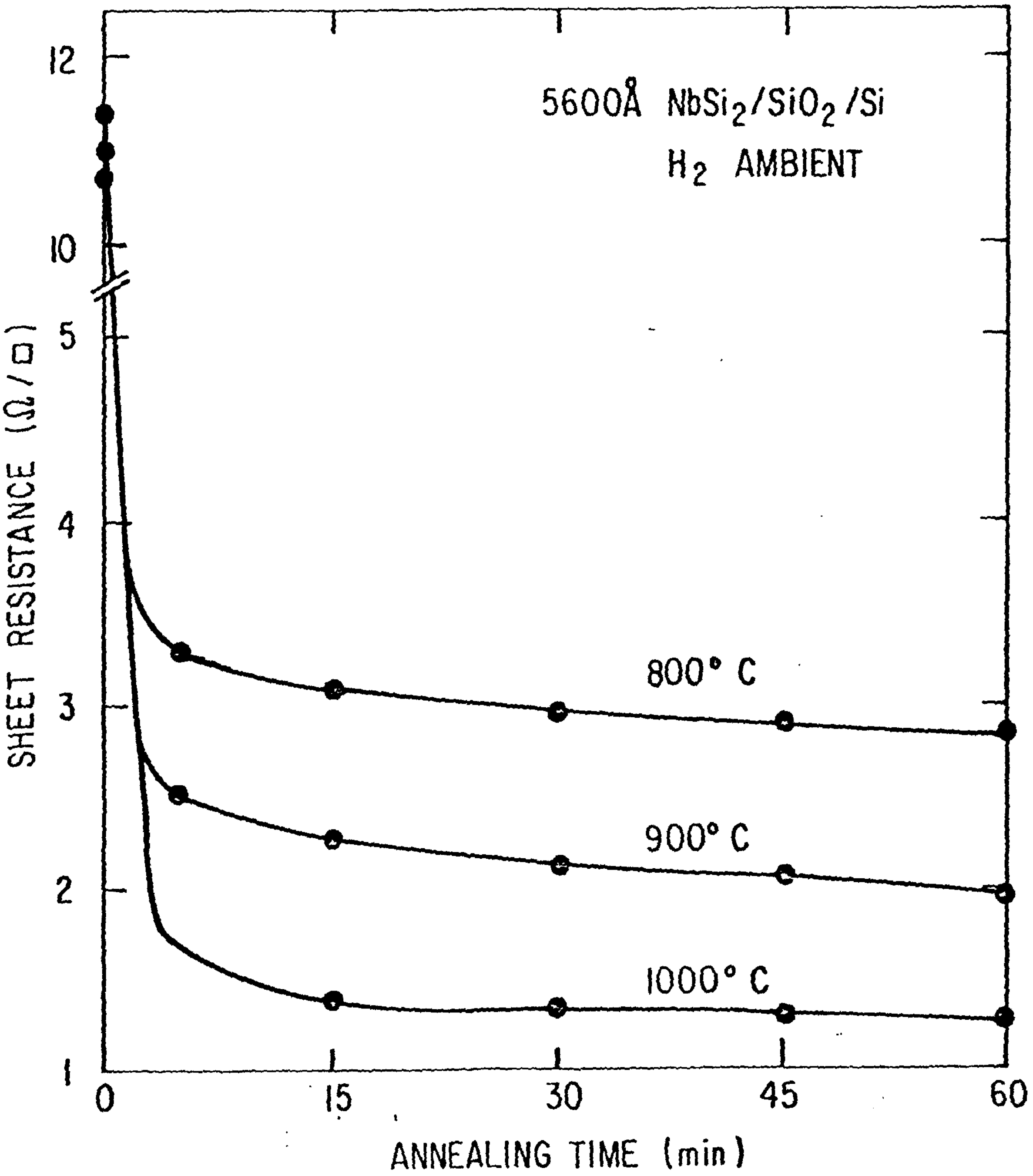
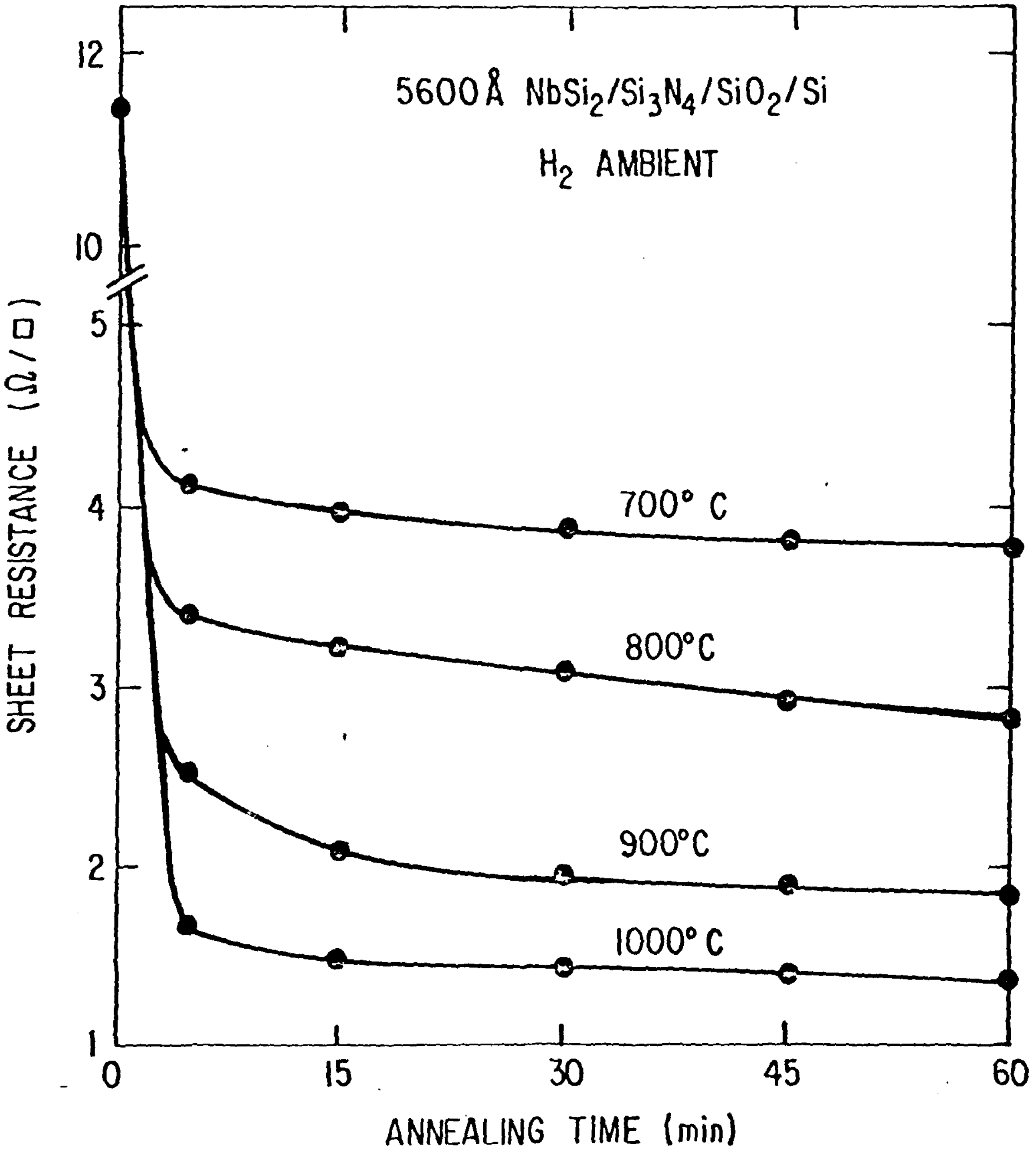
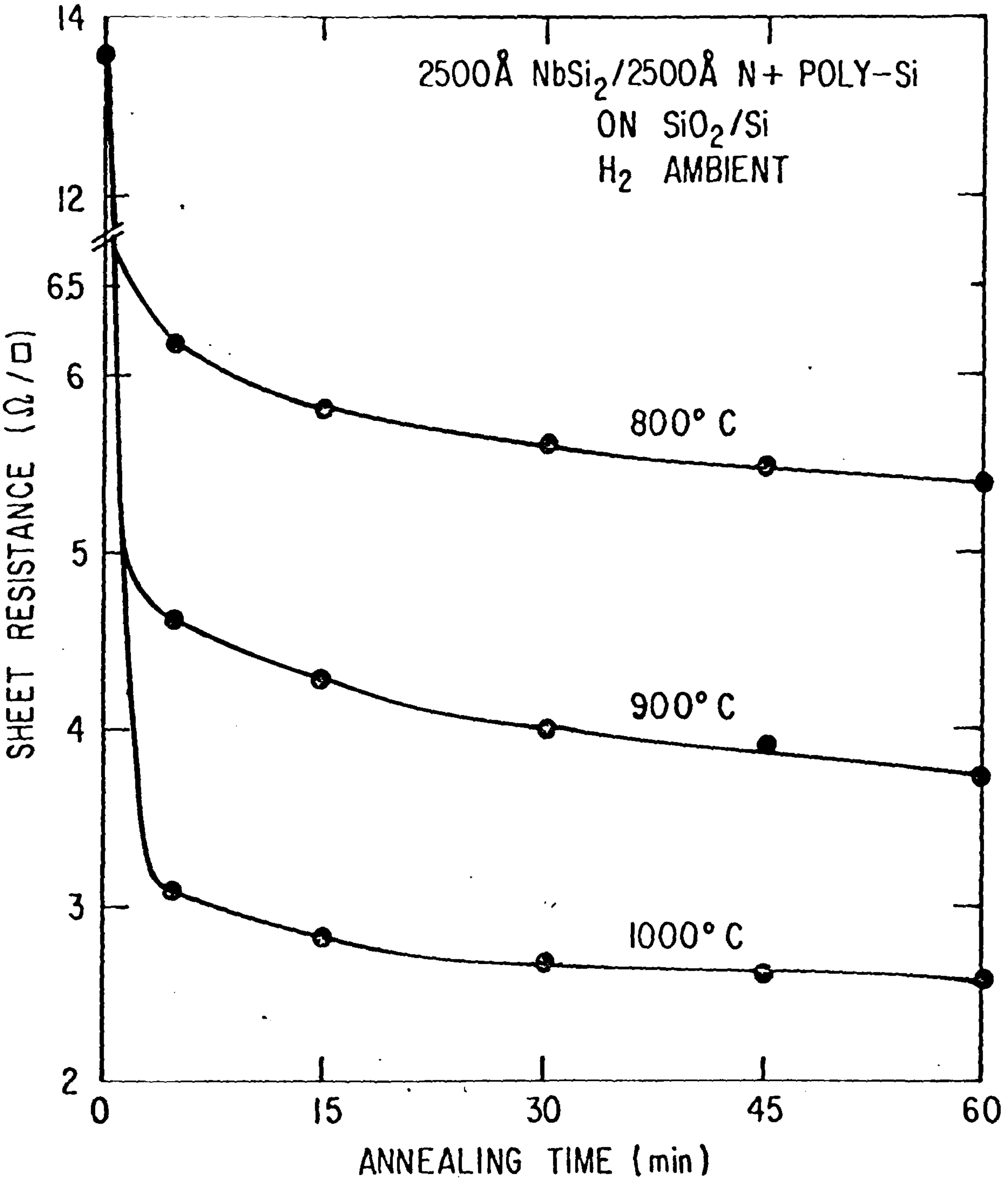
29
30
31
32
33
34

Si/Nb ~ 2.3



5600Å $NbSi_2/SiO_2/Si$
H_2 AMBIENT
SHEET RESISTANCE (Ω/□)
12
10
5
4
3
2
1
800° C
900° C
1000° C
0
15
30
45
60
ANNEALING TIME (min)


5600 Å $NbSi_2/Si_3N_4/SiO_2/Si$
H_2 AMBIENT
SHEET RESISTANCE (Ω/□)
12
10
5
4
3
2
1
700° C
800°C
900°C
1000° C
0
15
30
45
60
ANNEALING TIME (min)


2500Å $NbSi_2$/2500Å N+ POLY-Si
ON SiO_2/Si
H_2 AMBIENT
800° C
900° C
1000° C
SHEET RESISTANCE (Ω/□)
14
12
6.5
6
5
4
3
2
0
15
30
45
60
ANNEALING TIME (min)

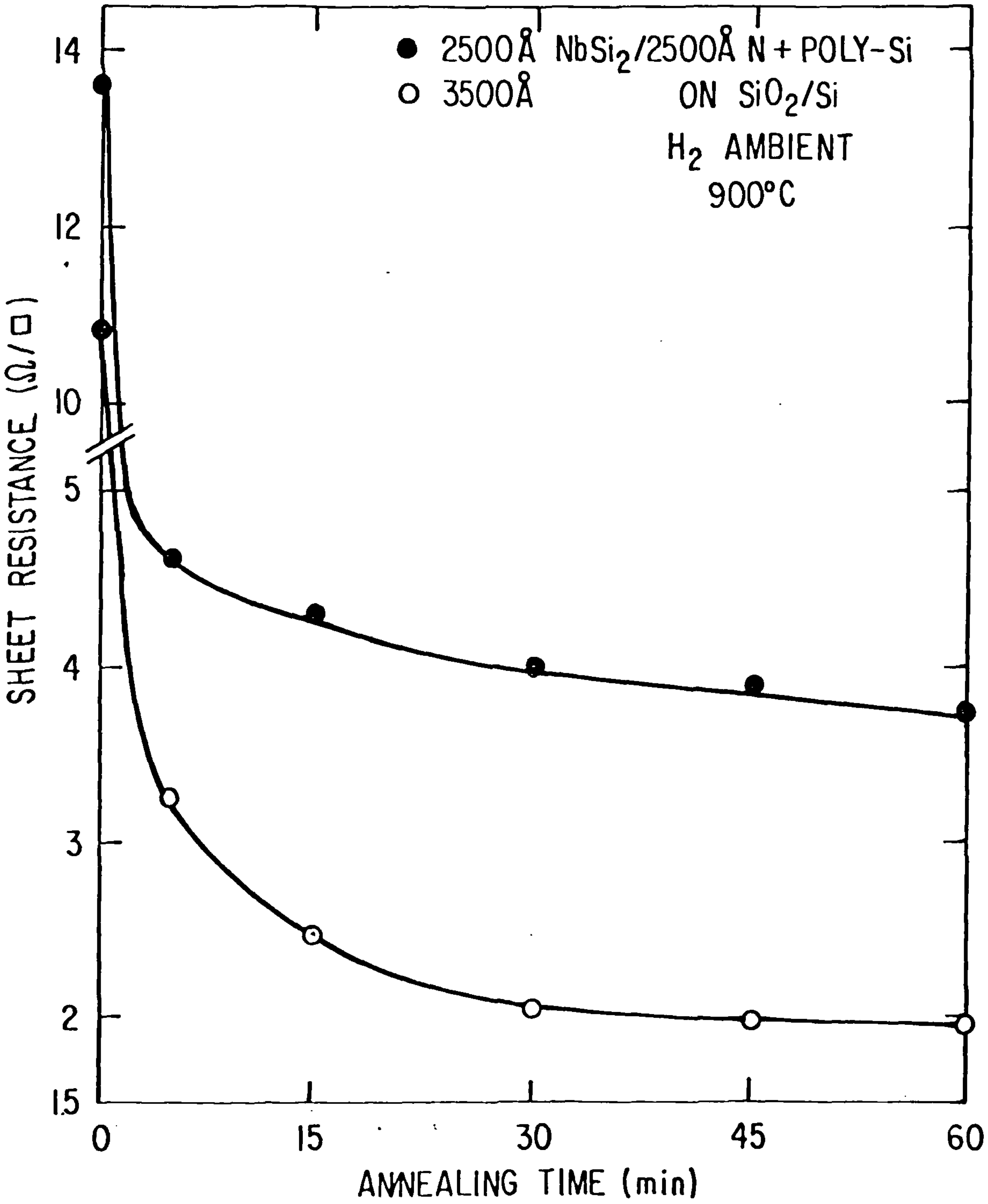

● 2500Å NbSi$_2$/2500Å N + POLY-Si
○ 3500Å
ON SiO$_2$/Si
H$_2$ AMBIENT
900°C
SHEET RESISTANCE (Ω/□)
14
12
10
5
4
3
2
1.5
0
15
30
45
60
ANNEALING TIME (min)

Table I

Various silicide phases that are formed for Group IV, V and VIA metals

IV A	V A	VI A
Ti_3Si	V_3Si	Cr_3Si
		Cr_2Si
	V_5Si_3	Cr_5Si_3
$Ti_5Si_3(C)$	$V_5Si_3(C)$	$Cr_5Si_3(C)$
		Cr_3Si_2
Ti_5Si_4	V_5Si_4	
	V_6Si_5	
$TiSi$		$CrSi$
fco-$TiSi_2$	VSi_2	$CrSi_2$
bco-$TiSi_2$		
Zr_4Si	Nb_4Si	
Zr_3Si	Nb_3Si	Mo_3Si
Zr_2Si		
	α-Nb_5Si_3 ($D8_1$)	Mo_5Si_3
	β-Nb_5Si_3 ($D8_m$)	
$Zr_5Si_3(C)$	$Nb_5Si_3(C)$	$Mo_5Si_3(C)$
Zr_3Si_2		Mo_3Si_2
Zr_4Si_3		
Zr_5Si_4		
Zr_6Si_5		
$ZrSi$		
$ZrSi_2$	$NbSi_2$	h-$MoSi_2$
		t-$MoSi_2$
	$Ta_{4.5}Si$	
	Ta_4Si	
	Ta_3Si	W_3Si
Hf_2Si	Ta_2Si	
	Ta_5Si_3	W_5Si_3 ($D8_m$)
	α-Ta_5Si_3 ($D8_1$)	
	β-Ta_5Si_3 ($D8_m$)	
$Hf_5Si_3(C)$	$Ta_5Si_3(C)$	$W_5Si_3(C)$
Hf_3Si_2		W_3Si_2
Hf_4Si_3		
Hf_5Si_4		
$HfSi$		
$HfSi_2$	$TaSi_2$	h-WSi_2
		t-WSi_2

Table II

Impurity contents of NbSi$_2$ films sputtered from hot-pressed and cold-pressed targets as determined from RBS and SIMS.

	Hot-Pressed	Cold-Pressed	Hot-Pressed/Cold-Pressed
C	1.8	1.0	1.8
O	8	10	0.8
Ar	20	8	2.5
SiN	25	5.5	4.5
(From SIMS)			
Si/Nb	1.7-1.8	2.1	
	2.0(Target)	2.3(Target)	
Ar	3%	3%	
Ta	0.2%	0.2%	
(From RBS)			
Target Purity	99.6%	99.6%	