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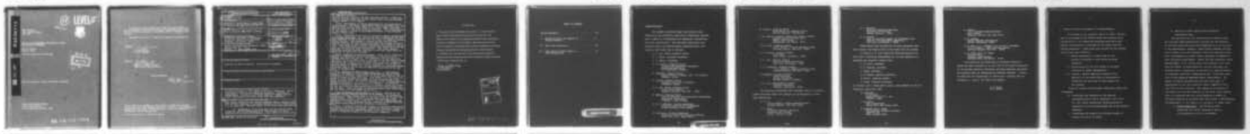

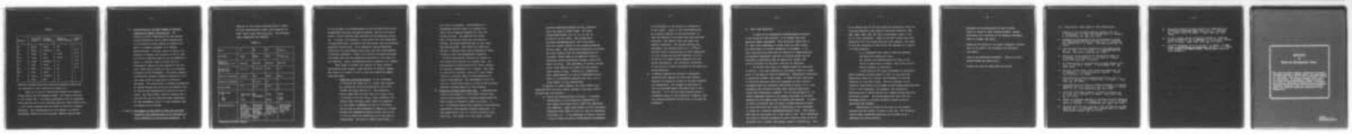

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ANALYSIS OF SEMICONDUCTOR STRUCTURES BY NUCLEAR AND ELECTRICAL TECHNIQUES

Marc-A. Nicolet
James W. Mayer

California Institute of Technology

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ROME AIR DEVELOPMENT CENTER
Air Force Systems Command
Griffiss Air Force Base, New York 13441

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Silicide formation of thin metal films on Si substrates. Marker experiments. Influence of substrate structure. Influence of contaminants. Stability of silicide layers in contact with Al films. Reaction of thin metal films with SiO_2 substrate.

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report summarizes the results obtained under a contract from 1974 to 1977, focusing on silicide formation of thin metal films on a Si substrate. The main thrust of the effort was directed at:

(i) Development of the data base on thin-film silicide formation, and the investigation of the influence of the Si substrate on the silicide formation. When taken in context with

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20. Abstract (cont'd)

results of other studies, the data obtained exhibit a clear pattern of behavior among the various metal films, but the detailed picture appears to be complex.

(ii) Development of marker experiments by ion-implanted Ar or Xe markers. The marker experiments with an implanted inert gas such as Ar or Xe have been shown to be a most useful technique for the study of thin-film reaction generally. The interpretation of the results in terms of microscopic features of the silicide remains largely open.

(iii) Clarification of the role played by oxygen contamination in silicide formation. The subject has been investigated for the PtSi formation at 600°C because this is a part in common Si device metallizations. When oxygen is present during the silicide-forming reaction, an SiO_2 layer grows below the surface of the Pt film as the front of the silicide approaches the surface of the Pt film. This oxide forms a barrier between the remaining Pt and the PtSi and the reaction stops prematurely. This layer imparts desirable apparent chemical inertness to the PtSi layer which is exploited in industrial processes.

(iv) Stability of silicide layers. Pd_2Si can be stabilized against an overlain Al film by interposing a thin film of Cr between the Pd_2Si and the Al. The protective action comes from the fact that Cr on Pd_2Si forms a silicide of its own ($CrSi_2$) under annealing, but without that the Pd_2Si layer loses its integrity. Cr also forms a compound with the Al film above, but as long as some unreacted Cr remains as a buffer between the compounds formed on either side by the Cr film an effective separation of Al and Pd_2Si is accomplished.

(v) Reaction of metal layers with SiO_2. A comprehensive study of the behavior of thin metal films on SiO_2 during vacuum annealing at elevated temperatures has established that Hf, Nb, Ti, V and Zr actually react with SiO_2. The result is a thin layer of metal silicide sandwiched between the SiO_2 substrate and a top layer of metal oxide. The results conform to the thermodynamically predicted outcome on the basis of that reaction. An empirical method to determine without calculation whether a metal will react or not with SiO_2 has been established using the electronegativity of the metal. This knowledge is valuable because metal films which do not react with SiO_2 adhere poorly to it.

(vi) Electrical characteristics of Pd_2Si. Because of its extended use as a contact material in Si devices, the electrical properties of Pd_2Si formed from a thin film of Pd and Si have been investigated. Pd_2Si is metallic in character and has a bulk electron concentration of $4 \times 10^{21} cm^{-3}$. At room temperature, the resistivity is about $30 \mu\Omega cm$ and the Hall mobility is about $50 cm^2/Vs$, but these values depend on the substrate orientation.

(vii) A computer program was written to synthesize backscattering spectra for thin-film samples composed of successive layers of uniform thickness and composition. It is the only such program which is accessible openly to the scientific community.

EVALUATION

1. This report is the Final Report on the contract. It covers research done on metal silicide formation during the three year period. The objective of the research is the understanding and control over metallization processes in integrated circuit processing. Many novel as well as conventional metallization schemes have been studied and significant contributions have been made towards the understanding of metal silicide formation.

2. The above work is of value since it provides basic knowledge which makes possible new and improved devices for accomplishing signal processing in USAF radar, communications, etc.

D. EIRUG DAVIES
Project Engineer

TABLE OF CONTENTS

ACKNOWLEDGEMENTS

The studies performed under this contract were executed at the California Institute of Technology together with a number of collaborators at industrial laboratories and other universities. The following individuals have authored and/or co-authored papers published under this contract while they were at Caltech:

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J. W. Mayer
M-A. Nicolet

I. Review of Goals and Summary of Accomplishments.

At the date of its inception (early in 1974), the main goal to be pursued by the studies under this contract was a clarification of the causes and processes which drive and control the formation of silicides by thin metal films on silicon substrates. Three areas were singled out for studies in the first year (1974-75):

i Marker experiments, to shed light on the atomistic processes at work during silicide formation.
ii Clarification of the role played in silicide formation by oxygen contamination.
iii Develop a general empirical picture on the behavior of thin metal films on substrates by broadening the data base to other metals not yet studied.

Along the course of the program, additional topics were introduced:

iv Investigate the influence of the physical characteristics of the Si substrate (<111>,<110> or <100> single crystalline, polycrystalline or amorphous) on the silicide phases and on the kinetics observed.
v Investigate the stability of silicide layers in contact with Al or Au films.

vi Reaction of metal layers with dielectrics, particularly SiO_2.

The motivation for all these choices is derived from the practical significance which silicides and the formation of silicides from thin metal films deposited on Si or SiO_2 have in the semiconductor industry. Metallization schemes utilized in the industry were and frequently still are relatively crude. Their understanding is almost always inadequate as compared to their practical significance. Surely the most noteworthy single accomplishment of our efforts under this contract is the fact that the subject of metallization on silicon (and SiO_2) has obtained general recognition as one which is indeed accessible to successful analytical investigation and, in the best cases, even to some adequate phenomenological understanding. The degree of success obtained over the last three years is demonstrated by the last Scientific Report (RADC-TR-77-254, July 1977) of this contract. That report is an overview of the subject of silicide formation by thin metal films reacting with a Si substrate and will be published as a chapter on silicide formation in an edited book "Thin Films - Interdiffusion and Reactions", (J. M. Poate, K. N. Tue and J. W. Mayer, Eds.).

i) Marker Experiments. The following table summarizes the results obtained to date by ion implantation of Ar or Xe markers:

TABLE 1

Element	Silicide Formed	Crystal Structure	Dominant Moving Specie	Growth Rate
Mg	Mg_2Si	cubic	Mg	?
Ni	Ni_2Si	orthorh.	Ni	$t^{1/2}$
Pd	Pd_2Si	hex.	Pd,Si	$t^{1/2}$
Pt*	Pt_2Si	tetr.	Pt,Si	$t^{1/2}$
Co*	Co_2Si	orthorh.	Co	$t^{1/2}$
Fe	FeSi	cubic	Si	$t^{1/2}$
Hf*	HfSi	orthorh.	Si	$t^{1/2}$
Rh*	RhSi	cubic	Si	?
Ti	$TiSi_2$	orthorh.	Si	?
V	VSi_2	hex.	Si	?

*Results included here from studies performed by others and not supported by this contract are marked by *.

There is no obvious correlation between the dominant moving specie and the compound formed, its crystal structure or the growth rate. The marker experiments with an implanted inert gas such as Ar or Xe has been shown by these results to be a most useful technique for the study of thin-film reaction generally. The interpretation of the results, in terms of microscopic features of the silicide, remains largely open.

ii) Clarification of the Role Played in Silicide Formation by Oxygen Contamination. The subject has been investigated for the platium silicide formation PtSi at 600°C because this is the stable silicide against an excess of Si and is a frequent component in Si device metallizations. The main result is that in the presence of oxygen, the reaction of the Pt film with the Si substrate does not go to completion. Rather, a residual layer of about 160 Å of unreacted Pt remains on the surface of the PtSi film, below which a layer of SiO_2 of an average thickness of 400 Å develops. This layer imparts the desirable apparent chemical inertness of the PtSi layer which is exploited in industrial processes. The oxide layer is caused by permeation of oxygen through the Pt film as the growth front of the silicide approaches the surface of the Pt films. The oxide forms a barrier between the Pt and the PtSi and the reaction stops prematurely. No such phenomenon occurs in inert ambients such as vacuum or dry nitrogen.

iii and iv) Development of Data Base on Thin-Film Silicide Formation, and Investigation of the Influence of the Si Substrate on the Silicide Formation. In addition to the studies reported under i) above, we have investigated in detail the formation of FeSi, Pd_2Si, Ni_2Si and $Ti_xW_{1-x}Si_2$. The following table summarizes the findings:

TABLE II

Metal	Ni	Pd	Fe	Ti_xW_{1-x}
Silicide	Ni_2Si	Pd_2Si	FeSi	$Ti_xW_{1-x}Si_2$
Range of Temperature (°C)	200-325	260-550	450-527	700-750
Activation Energy (eV)	1.3-1.6	1.3-1.5)*	1.67	4.5
Crystal Structure	orthorh.	hex.	cubic	hex.
Dominant Diffuser	Ni	Pd & Si	Si	
Growth Rate	$t^{1/2}$	$t^{1/2}$	$t^{1/2}$	t
Growth Rate				
Hi	(100), poly, am	no influence	(111)	(100)
Med				(110)
Low	(111)		(100)	(111)
Microstructure	Random gains of ~600 Å size on (100), oriented grains of ~1300 Å size on (111).	Epitaxial on (111); oriented grains columnar on (100) of 150 Å size	Poly-crystal, untextured for both (111) and (100).	Polycrystal. untextured for (100), (110) and (111).

*measured by other authors.

As can be seen, the microstructure of the silicide films varies considerably from one silicide to another, and can also differ within a given silicide upon variation of the substrate structure. In Ni_2Si, for instance, it was shown that this difference is associated with a different silicide growth rate for different substrate orientations. On an amorphous (vacuum deposited) substrate, one even observes both Ni_2Si and NiSi simultaneously in distinct sublayers. There is no visible correlation between the growth rate and the substrate orientation or the nature of the kinetics. Again, the detailed picture appears to be complex.

When taken in context with results of other studies performed by other groups, clear patterns of behavior appear. The subject is treated in full in the last Scientific Report mentioned above.

v. Stability of Silicide Layers. We have succeeded in assuring the stability of a Pd_2Si layer against an overlain Al film by interposing a film of Cr between the Pd_2Si and the Al. The protective action comes from the fact that Cr on Pd_2Si forms a silicide of its own ($CrSi_2$) under annealing, but without that the Pd_2Si layer loses its integrity. Cr also forms a compound with the Al film above, but as long as some unreacted Cr remains as a buffer between the compounds formed on either side by the Cr film an effective separation of Al and Pd_2Si is accomplished. The barrier effect breaks down if all the Cr is consumed. This concept of a consumeable barrier layer is a general one, and it can be applied whenever two main conditions are met: the reactions on either side of the barrier layer must be laterally uniform, and these reactions must be well known and characterized. Once these two conditions are met, a barrier layer can be designed on paper to meet a given time-temperature stressing.

We have successfully tested this concept of the "sacrificial barrier" on another case: the stablization of polycrystalline Si layers against Al films by interposed Ti or V layers. This case is practically relevant, since Al is used commercially as a contact layer to polycrystalline Si gates of MQS devices. The development of the concept of a sacrificial barrier is one of the prominent practical accomplishments of this contract.

vi <u>Reaction of Metal Layers With SiO_2</u>. A comprehensive study of the behavior of thin metal films on SiO_2 during vacuum annealing at elevated temperatures has been brought to completion under this grant. In all, 13 different metals were studied by backscattering spectrometry and scanning electron microscopy. It was found that Hf, Nb, Ti, V and Zr actually react with SiO_2. The result is a thin layer of metal silicide sandwiched between the SiO_2 substrate and a top layer of metal oxide. The other investigated metals (Co, Cr, Cu, Fe, Mu, Ni, Pd and Pt) do not react. The systems were analyzed thermodynamically, and it was found that the results conform to the outcome predicted on that basis. The theoretical prediction correlates well and linearly with the electronegativity of the metal, which leads to an empirical method to determine without calculation whether a metal will react with SiO_2. This knowledge is valuable because metal films which do not react with SiO_2 adhere quite poorly to SiO_2. Chromium is a borderline case, experimentally as well as thermodynamically: there is no detectable reaction with SiO_2, but Cr films adhere to SiO_2 nevertheless.

In addition to these results, two other studies were completed which are both closely related to the main efforts pursued under the contract:

a) Electrical Characteristics of Pd_2Si. A thorough investigation of the electrical conductivity of thin Pd_2Si films from 4.2°K to 320°K was undertaken and completed. Pd_2Si films differ in their structural properties when grown on (111) or (100) Si substrates (see Table II). It was therefore of general interest to see if these structural differences are accompanied by differences in the electrical conductivity of the layers. Pd_2Si is also used extensively as a contact material in silicon devices, so that the results of the study also have practical relevance. The results show that Pd_2Si has metallic character. The charge carriers are electrons, with concentration of $4 \times 10^{21} cm^{-3}$. The bulk value of resistivity at room temperature is 25 to 30 μΩcm and the Hall mobility is 50 to 60 cm^2/Vs, but these values depend on the substrate orientation. The structural difference of the films is clearly reflected by the electrical characteristics.

b) A computer program was written to synthesize backscattering spectra for thin-film samples composed of successive layers of uniform thickness and composition. The program is contained in Interim Technical Report RADC-TR-76-182 of this contract. It is the only such program which is accessible openly to the scientific community, and has rendered excellent service both at Caltech and elsewhere.

II. Major Open Questions.

In spite of the substantial accomplishments obtained in the course of this contract, it is evident that many questions remain unanswered. This conclusion holds even if all other work undertaken in recent years by other groups and individuals on silicide formation is included. It is clear that much more work will be required to understand both the physical mechanisms involved in silicide growth and the influence of parameters such as impurities, stress, and initial structure of the metal film and the substrate.

We believe that there is a deeper reason for this state of affairs. Silicide formation by thin metal films is a novel topic of the general field of metallurgy. Metallurgy is basically an empirical science, even today. It would be most surprising if thin-film reactions would be accessible to more rigorous treatments than the scientific discipline of which it is a part. Increasing the surface-to-volume ratio usually complicates rather than simplifies a problem. Out of that perspective, the largely empirical status of our present knowledge of silicide formation by thin metal films is natural and expected. It would be wrong to conclude that the study of these reactions should be laid aside until the field of metallurgy of bulk phenomena is placed on a rigorous nonphenomenological basis. That develoment may not take place for a long time to come. More importantly, the study of silicide formation by thin films may prove to be an excellent way to further the general subject of metallurgy. This is so because many of the most sensitive analytical tools now currently applied to the study of thin-film reactions (TEM, LEEP, REED, SIMS, AES, BS) have a microscopic range in one, two or three dimensions. They are by their nature much more compatible with thin films than with bulk samples. The study of silicide formation by thin films thus emerges as a subject of unique standing:

- it is compatible with some of the most advanced and sensitive analytical tools.
- the results are simultaneously of value to the field of metallurgy in general, and to the silicon device industry in particular.
- it can build on the highly developed Si technology.

These arguments strongly argue in favor of continued and substantive efforts to further the knowledge of silicide formation by thin metal films. When the interest of the industrial application will broaden to more complicated materials (III-V, IV-VI compounds, for example), the problems of metallurgy and of metallization will rapidly become much more difficult to grasp. One would expect that here also the knowledge gained in silicide formation should provide a background and guidance.

Specifically in terms of the goals of the present contract and the results to which it has led, the following list of major unanswered questions can be drawn up as a reference for future efforts:

- **Determine the microstructure of the silicide formed in terms of their crystallography, texture and defects as a function of the external variables. (What is formed, and when?).**
- **Establish the nature of the atomic transport involved. (Why is the growth rate different for different silicides?).**
- **Clarify the nucleation processes. (Why are certain phases formed and others not?).**
- **Clarify the role of impurities and stress.**

III. Publications (Under AFCRL or RADC Sponsorship).

1. Studies of the Ti-W Metallization System on Si, by J. M. Harris, S. S. Lau, M-A. Nicolet and R. S. Nowicki. J. Electrochem. Soc. 123, 120 (1976).

2. Influence of the Nature of the Si Substrate on Nickel Silicide Formed From Thin Ni Films, by J. O. Olowolafe, M-A. Nicolet and J. W. Mayer. Thin Solid Films 38, 143 (1976).

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7. Investigation of Tellurium-Implanted Silicon, by T. F. Lee, R. D. Pashley, T. C. McGill and J. W. Mayer. J. Appl. Phys. 46, 381 (1975).

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13. Ti and V Layers Retard Interaction Between Al Films and Polycrystalline Si, by K. Nakamura, S. S. Lau, M-A. Nicolet and J. W. Mayer. Appl. Phys. Lett. 28, 277 (1976).

14. Silicide Formation, by K. N. Tu and J. W. Mayer. in Thin Films - Interdiffusion and Reactions, Edited by J. M. Poate, K. N. Tu and J. W. Mayer (Wiley - Interscience, New York, 1978) Chapter 8.

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