

**PUSHKAR JAIN** 

# Tantalum Oxide Thin Films for Embedded Capacitors

**Using Pulsed DC Reactive Sputtering** 



# **PUSHKAR JAIN**

# Tantalum Oxide Thin Films for Embedded Capacitors

**Using Pulsed DC Reactive Sputtering** 

VDM Verlag Dr. Müller

Impressum/Imprint (nur für Deutschland/ only for Germany)

Bibliografische Information der Deutschen Nationalbibliothek: Die Deutsche Nationalbibliothek verzeichnet diese Publikation in der Deutschen Nationalbibliografie; detaillierte bibliografische Daten sind im Internet über <a href="http://dnb.d-nb.de">http://dnb.d-nb.de</a> abrufbar.

Alle in diesem Buch genannten Marken und Produktnamen unterliegen warenzeichen-, markenoder patentrechtlichem Schutz bzw. sind Warenzeichen oder eingetragene Warenzeichen der
jeweiligen Inhaber. Die Wiedergabe von Marken, Produktnamen, Gebrauchsnamen,
Handelsnamen, Warenbezeichnungen u.s.w. in diesem Werk berechtigt auch ohne besondere
Kennzeichnung nicht zu der Annahme, dass solche Namen im Sinne der Warenzeichen- und
Markenschutzgesetzgebung als frei zu betrachten wären und daher von jedermann benutzt
werden dürften.

Coverbild: www.purestockx.com

Verlag: VDM Verlag Dr. Müller Aktiengesellschaft & Co. KG Dudweiler Landstr. 125 a, 66123 Saarbrücken, Deutschland

Telefon +49 681 9100-698, Telefax +49 681 9100-988, Email: info@vdm-verlag.de

Zugl.: New York, Rensselaer Polytechnic Institute, PhD Dissertation, 2003

Herstellung in Deutschland:

Schaltungsdienst Lange o.H.G., Zehrensdorfer Str. 11, D-12277 Berlin Books on Demand GmbH, Gutenbergring 53, D-22848 Norderstedt Reha GmbH, Dudweiler Landstr. 99, D-66123 Saarbrücken

ISBN: 978-3-639-09662-0

Imprint (only for USA, GB)

Bibliographic information published by the Deutsche Nationalbibliothek: The Deutsche

Nationalbibliothek lists this publication in the Deutsche Nationalbibliografie; detailed bibliographic data are available in the Internet at <a href="http://dnb.d-nb.de">http://dnb.d-nb.de</a>.

Any brand names and product names mentioned in this book are subject to trademark, brand or patent protection and are trademarks or registered trademarks of their respective holders. The use of brand names, product names, common names, trade names, product descriptions etc. even without

a particular marking in this works is in no way to be construed to mean that such names may be regarded as unrestricted in respect of trademark and brand protection legislation and could thus be used by anyone.

Cover image: www.purestockx.com

#### Publisher:

VDM Verlag Dr. Müller Aktiengesellschaft & Co. KG Dudweiler Landstr. 125 a, 66123 Saarbrücken, Germany Phone +49 681 9100-698, Fax +49 681 9100-988, Email: info@vdm-verlag.de

Copyright © 2008 VDM Verlag Dr. Müller Aktiengesellschaft & Co. KG and licensors All rights reserved. Saarbrücken 2008

Produced in USA and UK by:

Lightning Source Inc., 1246 Heil Quaker Blvd., La Vergne, TN 37086, USA Lightning Source UK Ltd., Chapter House, Pitfield, Kiln Farm, Milton Keynes, MK11 3LW, GB BookSurge, 7290 B. Investment Drive, North Charleston, SC 29418, USA ISBN: 978-3-639-09662-0

# **PUSHKAR JAIN**

**Tantalum Oxide Thin Films for Embedded Capacitors** 

#### ACKNOWLEDGMENT

First and foremost, I would like to acknowledge and thank my advisor Prof. E. J. Rymaszewski and Prof. T. S. Cale for their guidance and relentless support without which this thesis would not have been possible. Their knowledge, guidance, and support were crucial for the completion of this work. I would also like to acknowledge Prof. T. –M. Lu for guiding me like an advisor, and encouraging me to try out new ideas. I would like to thank my other committee members, Prof. R. J. Gutmann, and Prof. R. Kane for their guidance and valuable advice. The constructive criticism and guidance of my committee members helped me focus better on the thesis work.

I would also like to acknowledge Interconnect Focus Center and Center for Power Electronics Systems for providing me the financial support and research funding in various phases of this work.

Throughout this work, I have been helped by numerous other people in various capacities. I would like to thank Jasbir and Vinay, who helped me out tremendously throughout this work. Thanks are due to Anu and Tansel for sharing their immense experience in metal ion penetration and roughness evolution studies, respectively. I would also like thank Ravi and Jasbir for thoroughly reviewing this thesis, and providing valuable input in shaping this thesis manuscript. The clean room and the administrative staff at the Center for Integrated Electronics have been very helpful during my stay at RPI. I would like to thank Dana, Kelli, and Joelle for their help. My stay at Troy has been enjoyable because of the many great friends I made here. I would like to acknowledge Tuhin, Anju, Shom, Jasbir, Ravi, Amrit, Ashish, Rohit, Anu, Manisha, Tansel, Yongchai, Suchira, and Ed.

Last, but not the least, I would like to thank my family members for their love, support, and encouragement throughout the course of my academic career.

xviii

#### ABSTRACT

Embedded capacitor technology, where thin film capacitors are integrated at on-chip and/or off-chip levels, offers high packaging densities and improved electrical performance at potentially reduced costs of capacitor fabrication and integration. This research explores and establishes the leverages of using thin film embedded capacitors over currently used surface mount discrete capacitors. In particular, this thesis focuses on developing pulsed dc reactively sputtered tantalum oxide (Ta<sub>2</sub>O<sub>5</sub>) thin film capacitors to be integrated into established interconnect technologies of IC chips and packages.

A correlation between electrical breakdown field and dielectric constant,  $E_{BR}$  (MV/cm) =  $\left(20/\sqrt{\varepsilon_r}\right)$  is empirically determined and used to establish a design space for breakdown voltage and capacitance density of planar capacitors, with film thickness and material dielectric constant as parameters. This design space sets the limits for "best one can achieve" (BOCA) breakdown voltages and capacitance densities using a particular dielectric. The validity of the developed design space is experimentally verified with  $Ta_2O_5$  thin films over a wide range of film thickness (0.05 to 5.4 µm). Detractors causing the deviations from the BOCA breakdown voltages are identified and corrected experimentally. In particular, substrate cooling during deposition of "thicker" (> ~1 µm) films is required to deposit stoichiometric  $Ta_2O_5$  exhibiting BOCA breakdown voltages, and "peak-to-valley" roughness at the metal/dielectric interfaces must be less than 10% of dielectric film thickness to avoid pre-mature breakdown of thin film capacitors.

An experimentally verified analytical model for pulsed dc reactive sputtering of Ta<sub>2</sub>O<sub>5</sub> films is described and evaluated. The influences of important process variables, like oxygen flow rate and sputtering ion current, on the oxygen partial pressure in the chamber, deposition rate, film stoichiometry as well as film breakdown and leakage

characteristics are predicted using this model. The experimentally established existence of multiple oxygen partial pressures at a given oxygen flow rate (hysteresis loop) is theoretically explained using steady state analysis. The experimental results suggest that in order to ensure the electrical reliability of Ta<sub>2</sub>O<sub>5</sub> films, deposition should be done at oxygen flow rates more than that required to go beyond the hysteresis region in oxygen partial pressure versus flow rate curve.

High frequency test vehicles were designed and fabricated to evaluate the electrical performance of Ta<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, and Si<sub>3</sub>N<sub>4</sub> thin film capacitors over a wide range of frequencies (dc to 20 GHz). Ta<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub>, and Si<sub>3</sub>N<sub>4</sub> show no dispersion at least up to 20 GHz. The total inductance of power connect vias is determined to be less than 50 pH/μm of via, which is at least two orders of magnitude lower than most discrete capacitors along with connection leads (> 4 nH). Providing several vias in parallel further reduces the overall inductance of the connection paths in thin film capacitors. The shorter connections and elimination of connection leads in thin film decoupling capacitors make them highly suitable for integration into 2D and 3D structures. Thin film capacitors based on SiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub>, and Ta<sub>2</sub>O<sub>5</sub> can provide capacitance densities comparable to the state-of-the-art surface mount discrete decoupling capacitors (~ 30 nF/cm<sup>2</sup>). The leakage current densities below 10<sup>-6</sup> A/cm<sup>2</sup> (at 0.5 MV/cm) are achieved with SiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub> and Ta<sub>2</sub>O<sub>5</sub> thin film dielectrics.

The extent of Cu diffusion/drift into  $Ta_2O_5$  films is determined and compared with Al, Ta, and Ti at various biasing and temperature conditions using biastemperature-stress (BTS) and triangular voltage sweep (TVS) techniques. No Cu diffusion was detected at 150 °C at least till 0.75 MV/cm. While, Cu diffuses/drifts into  $Ta_2O_5$  at aggressive temperature conditions, i.e., at 300 °C was detected, a thin layer of Ti or Ta (~30 nm) acts as an excellent Cu diffusion barrier into  $Ta_2O_5$ .

# TABLE OF CONTENTS

			Page
LIST	LIST OF TABLES		
LIST	OF FIGU	URES	ix
ACK	NOWLE	DGEMENT	xviii
ABS	TRACT		xix
СНА	PTER 1		
INTE	RODUCT	TION	1
1.1	Current	Capacitor Technology and Trends	1
1.2	Integrat	tion of Thin Film Capacitors: Motivations and Leverages	2
1.3	Issues a	and Challenges	4
1.4	Thesis	Focus: Goals and Approach	8
	1.4.1	Choice of Dielectric Material	10
	1.4.2	Choice of Deposition Technique	10
	1.4.3	Approach	11
	1.4.4	Thesis Overview and Structure	12
1.5	Thesis	Overview and Structure Thesis Overview and Structure	15
1.6	References		17
CHA	PTER 2		
CAP	ACITOR	R TECHNOLOGY: PROCESSES AND MATERIALS	21
2.1	Thick a	and Thin Film Capacitor Technologies	21
	2.1.1	Thick Film Capacitor Technologies	21
	2.1.2	Thin Film Capacitor Technology	22
2.2	Materi	als Options for Dielectrics for Thin Film Capacitors	24
23	Thin F	ilm Denosition Techniques for Tantalum Oxide	27

			Page
	2.3.1	Chemical Vapor Deposition (CVD)	27
	2.3.2	Oxidation Techniques	29
	2.3.3	Atomic Layer Deposition (ALD)	30
	2.3.4	Sputtering Techniques	31
2.4	Reactive	Sputtering System Used in This Thesis Work	32
	2.4.1	System Configuration	32
	2.4.2	Biasing Scheme Used	33
2.5	Summar	y of the Chapter	35
2.6	Reference	ces	36
CHAP	TER 3		
PROC	ESS MO	DELING AND OPTIMIZATION OF REACTIVE	
SPUT	TERING	;	40
3.1	Chapter	Overview and Structure	40
3.2	Historica	al Background and Our Approach	41
3.3	Reactive	Sputtering Model for Ta <sub>2</sub> O <sub>5</sub> Deposition	43
3.4	Experim	ental Methodology	49
	3.4.1	Partial Pressure Measurements	49
	3.4.2	Current-voltage (I-V) Characteristics	50
3.5	Results a	and Discussion	52
	3.5.1	Determination of Steady-State Partial Pressure of Oxygen	52
	3.5.2	Multiple Steady States	53
	3.5.3	Stability of Steady States	57
	3.5.4	Sputtering Ion Current	58
	3.5.5	Deposition Rate	60
	3.5.6	Electrical Properties	61
	3.5.7	Hysteresis in Breakdown Characteristics	64

				Pag
3.6	Summa	ary and Cond	clusions	65
3.7	Refere	nces		67
CHA	PTER 4			
BRE	AKDOW	N VOLTA	GE VERSUS CAPACITANCE DENSITY	
AND	DESIG	N LIMITS		69
4.1	Chapte	r Overview	and Structure	70
4.2	Ideal T	hin Film Die	electrics: Theoretical Limits	70
4.3	Design	Limits for c	apacitors with Ta2O5 thin films	74
	4.3.1	Experime	ntal Methodology	74
	4.3.2	Experime	ntal Results and Discussion	76
4.4	Deviat	ion from "Be	est can achieve" Performance	77
	4.4.1	Breakdow	m Field and Breakdown Voltage	77
	4.4.2	Dielectric	Constant and Dielectric Loss	81
	4.4.3	Leakage (	Current Density	83
	4.4.4	Chemical	Composition	84
	4.4.5	Control o	Film Stoichiometry	88
		4.4.5.1	Substrate Temperature	88
		4.4.5.2	Oxygen Partial Pressure	89
		4.4.5.3	Stoichiometric Oxide with Substrate Cooling	91
	4.4.6	Improved	Electrical Properties with Substrate Cooling	92
	4.4.7	Lower Breakdown Field in Thinner Films (< 0.15 μm)		96
4.5	Summa	ary and Cond	clusions	101
4.6	Refere	nces		103

			Page
СНА	PTER 5		
BRO	AD BAN	ND ELECTRICAL CHARACTERIZATION/	
EVA	LUATI	ON OF THIN FILM CAPACITORS	105
5.1	Chapte	er Overview and Structure	105
5.2	Thin F	ilm Decoupling Capacitors	106
5.3	Test V	ehicle: Design and Fabrication	110
	5.3.1	Test Vehicle Design	110
	5.3.2	Test Vehicle Fabrication	114
5.4	Charac	cterization Methodology	115
5.5	Result	s and Discussion	116
	5.5.1	Dielectric Constant and Dielectric Loss	116
		5.5.1.1 Low frequency characterization	117
		5.5.1.2 High frequency characterization	118
	5.5.2	Total Series Inductance	128
	5.5.3	Leakage Current Density	131
	5.5.4	Capacitance Density and Breakdown Field	132
5.6	Summ	ary and Conclusions	134
5.7	Refere	ences	136
CHA	PTER 6		
ELE	CTROD	E METAL DIFFUSION IN TANTALUM OXIDE	138
6.1	Overv	iew of the Chapter	139
6.2	Exper	imental Methodology Used	139
	6.2.1	Metal-Insulator-Semiconductor (MIS) Capacitors: Structure	
		and Fabrication	139
	6.2.2	Capacitance-Voltage (C-V) Characteristics of MIS Capacitors	140
	6.2.3	Bias-Temperature Stress (BTS) Technique	142

				Pag
	6.2.4	Triangula	r Voltage Sweep (TVS) Technique	144
6.3	Results	and Discus	sion	146
	6.3.1	Al/ SiO <sub>2</sub> /	Si and Cu/ SiO <sub>2</sub> / Si Capacitors	146
	6.3.2	Al/Ta <sub>2</sub> O <sub>5</sub> /	SiO <sub>2</sub> /Si and Cu/Ta <sub>2</sub> O <sub>5</sub> /SiO <sub>2</sub> /Si Capacitors	148
		6.3.2.1	BTS Testing Results	148
		6.3.2.2	TVS Characterization Results	152
	6.3.3	Mobile C	harge (Cu ions) Calculations	154
		6.3.3.1	Mobile charges using BTS results	154
		6.3.3.2	Mobile charges using TVS results	155
	6.3.4	Measures	to Contain Cu-Diffusion	156
6.4	Summa	ry and Cond	clusions	160
6.5	Referen	ces		161
СНА	PTER 7			
SUM	MARY, (	CONCLUS	IONS, AND REMAINING CHALLENGES	163
7.1	Overall	Summary o	of the Thesis	163
	7.1.1	Optimizat	ion and Control of Reactive Sputtering Deposition of	
		$Ta_2O_5$		163
	7.1.2	Breakdow	n Voltage Versus Capacitance Density and Design	
		Limits		165
	7.1.3	Broadban	d Electrical Characterization/Evaluation of Thin	
		Film Capa	acitors	167
	7.1.4	Electrode	Metal Diffusion in Tantalum Oxide	168
7.2	Conclus	Conclusions 1		
7.3	Next Set of Challenges and Recommendations			171

		Page
APPENDIX A	Surface Roughness Growth In Reactively Sputtered	
	$Ta_2O_5$	174
APPENDIX B	Parameters Extraction From S <sub>21</sub> Measurements	183

# LIST OF TABLES

		Page
Table 1.1	Comparison of the number of active (ICs) and passive components	1
Table 2.1.	Dielectric constants of important paraelectric and ferroelectric	
	Dielectrics	24
Table 2.2.	Comparison of paraelectric and ferroelectric dielectrics	26
Table 3.1.	Steady-state oxygen partial pressures corresponding to the	
	different oxygen flow rates. Multiple steady states are realized	
	between $Q_{02}$ and $Q_{05}$ .	55
Table 3.2.	The values of the parameters used in the proposed analytical model	58
Table 4.1.	Atomic percent of tantalum and oxygen present in reactively	
	sputtered tantalum oxide films of different film thicknesses	87
Table 5.1.	Physical dimensions and number of vias present in the capacitors	
	of the test vehicle. Nominal capacitances and via inductances are	
	also listed.	111
Table 5.2.	Deposition conditions for the dielectrics used in the test vehicles	114
Table 6.1.	Metal ion penetration after 120 minutes of biasing	160

### LIST OF FIGURES

		Page
Figure 1.1	Photographs of IBM's multi-chip module showing the	(u)
	arrangement of IC chips surrounded by the decoupling capacitors,	(b)
	blown-up view of surface mount discrete decoupling capacitor, and	(c)
	IC and capacitor footprints [8].	3
Figure 1.2.	Schematics showing the integration of thin film capacitors (a) within	the
	substrate of the packaging module (off-chip integration) and (b) wit	hin
	the multilevel metallization (MLM) of the IC chip (on-chip integration	n). 4
Figure 1.3.	Schematic showing the footprints of the IC and decoupling capacit	tors
	along with the capacitor-to-IC connection path.	7
Figure 2.1.	Schematic of the reactive sputtering system used.	33
Figure 2.2.	Target poisoning occurs during reactive sputtering with a metallic target	get.
	Without the pulsing unit, severe target poisoning will result arcing	34
Figure 3.1.	Schematic showing the oxygen flow through the reactive sputter	ring
	system. $Q_0$ is the incoming gas flow of which $Q_{\text{target}}$ is consumed at	the
	target, $Q_{\text{chamber}}$ is consumed at the chamber walls and substrate and $Q_{\text{t}}$	pump
	goes out of the chamber through the pump.	44
Figure 3.2.	Schematic of particle fluxes during reactive sputtering. J is the sputter	ring
	ion current density. $F_1\text{-}F_4$ denote fluxes of sputtered material. $F_{O2}$ is	the
	flux of neutral oxygen molecules originating from the oxygen par	tial
	pressure $p_{O2}$ . Dotted regions represent the elemental tantalum ato	ms.
	Hatched regions represent tantalum oxide molecules.	45
Figure 3.3.	A schematic of pulsed dc reactive sputtering reactor used in this study	50
Figure 3.4.	(a) Biasing scheme used to measure dc leakage current density	and

breakdown field. (b) Top view of capacitor after electrical breakdown.

51

	states.	54
Figure 3.7.	Variation of steady-state oxygen partial pressure (p <sub>s</sub> )with oxygen flow	
	$rate(Q_0) \ for \ sputtering \ ion \ currents \ of: (a) \ 2 \ A, (b) \ 3 \ A, (c) \ 5 \ A, (d) \ 6 \ A.$	
	Solid lines (—) show the predicted curves using analytical model, ( $\Delta$ )	
	and (O) show the experimentally determined values for increasing and	
	decreasing flow rate modes respectively. Dotted lines () are the	
	experimental curves for I = 0 A (no sputtering) case.	59
Figure 3.8.	Experimental and predicted normalized deposition rates for I= 2A.	60
Figure 3.9.	Variation of breakdown field with oxygen flow rate in increasing flow	
	rate mode, for I= 2, 5 A. Dotted lines shows the predicted critical oxygen	
	flow rate using analytical model.	62
Figure 3.10.	Leakage current density of deposited $Ta_2O_5$ films at 0.5 MV/cm as a	
	function of oxygen flow rate, for sputtering ion current of 2 and 5 A.	63
Figure 3.11.	Breakdown field as a function of oxygen flow rate for sputtering ion	
	current of 5 A. Breakdown field shows hysteresis effect with respect to	
	oxygen flow rate during increasing and decreasing flow rate modes. The	
	position and the width of hysteresis in breakdown field is same as in	
	partial pressure curves.	64
Figure 4.1.	Breakdown field as a function of dielectric constant. Breakdown field	
	decreases as dielectric constant increases following $E_{BR}(MV/cm)$ =	
	$20/(\varepsilon_{\rm r})^{1/2}$ .	71

Steady-state oxygen partial pressure, ps at a given oxygen flow rate, Qo from the intersection of oxygen supply curve, "S" and oxygen

Determination of multiple steady states: (a) with the variation of oxygen flow rate,  $Q_0$  and (b) steady-state oxygen partial pressure( $p_s$ ) as a function of oxygen flow rate (Q0), showing upper, lower and unstable steady

53

54

Figure 3.5.

Figure 3.6.

consumption curve, "C".

Figure 4.2.	Design space with breakdown voltage and capacitance density as two	
	main figures of merit with film thickness and dielectric constant as	
	parameters. Solid lines are constant $\epsilon_{\text{r}}$ lines and dotted lines are constant	
	thickness lines. The targeted range of capacitance density needed for	
	next generation handheld systems are 13-70 nF/cm <sup>2</sup> [9,10].	73
Figure. 4.3.	Breakdown voltage as a function of capacitance density for tantalum	
	oxide thin films. The breakdown voltage increases from 10V to 900V	
	and capacitance density drops from 150 to 7 nF/cm <sup>2</sup> , when film-	
	thickness is increased from 0.05 to 5.4 $\mu m.$ Solid line (—) shows the	
	"best can achieve" values, solid dots (•) are the experimental values that	
	are in agreement with the "best can achieve" values, and hollow dots (o)	
	are the regions of significant deviations from the "best can achieve"	
	values.	76
Figure 4.4.	Breakdown field of tantalum oxide films as a function of film thickness,	
	showing a decline in breakdown field for "thinner" films (< $\sim\!\!0.15~\mu m)$	
	and also for "thicker" films (>~ 1 $\mu$ m).	78
Figure 4.5.	Variation of breakdown voltage with film thickness, showing the	
	deviation from the straight line corresponding to 3.5 MV/cm, for films	
	thicker than 1 $\mu m,$ and for films thinner than around 0.15 $\mu m.$	79
Figure 4.6.	Cumulative probability of failure due to electrical breakdown (y-axis)	
	below a particular breakdown voltage (x-axis) for films with thickness	
	from 0.12 to 5.4 $\mu m$ .	80
Figure 4.7	(a). Variation of apparent (or measured) dielectric constant with film	
	thickness. (b) Dielectric loss (measured) versus film thickness, showing	
	relatively higher loss for films thicker than around 1 $\mu m$ .	82
Figure 4.8.	Dependence of leakage current density on applied field for films with	
	thickness varying from 0.12 to 5.4 $\mu m$ .	83