Werkstoffanwendungen im Maschinenbau

Band 21

Sree Koundinya Sistla

Numerical Modelling and Simulation of Temperature Fields, Densification and Grain Growth during Field Assisted Sintering Technology





Institut für Anwendungstechnik Pulvermetallurgie und Keramik an der RWTH Aachen e.V.

Herausgeber: Prof. Dr.-Ing. C. Broeckmann

Numerical Modelling and Simulation of Temperature Fields, Densification and Grain Growth during Field Assisted Sintering Technology

Numerische Modellierung und Simulation von Temperaturfeldern, Verdichtung und Kornwachstum beim feldunterstützten Sintern

Von der Fakultät für Maschinenwesen der Rheinisch-Westfälischen Technischen Hochschule Aachen zur Erlangung des akademischen Grades eines Doktors der Ingenieurwissenschaften genehmigte Dissertation

vorgelegt von

Sree Koundinya Sistla

Berichter: Univ.-Prof. Dr.-Ing. C. Broeckmann Apl.-Prof. Dr. Martin Bram

Tag der mündlichen Prüfung: 21. Juni 2021

Diese Dissertation ist auf den Internetseiten der Universitätsbibliothek online verfügbar

Werkstoffanwendungen im Maschinenbau hrsg. von Prof. Dr.-Ing. Christoph Broeckmann

Band 21

Sree Koundinya Sistla

Numerical Modelling and Simulation of Temperature Fields, Densification and Grain Growth during Field Assisted Sintering Technology

> Shaker Verlag Düren 2021

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 http://dnb.d-nb.de.

Zugl.: D 82 (Diss. RWTH Aachen University, 2021)

Copyright Shaker Verlag 2021 All rights reserved. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording or otherwise, without the prior permission of the publishers.

Printed in Germany.

ISBN 978-3-8440-8173-2 ISSN 2195-2981

Shaker Verlag GmbH • Am Langen Graben 15a • 52353 Düren Phone: 0049/2421/99011-0 • Telefax: 0049/2421/99011-9 Internet: www.shaker.de • e-mail: info@shaker.de

Acknowledgment

This thesis was written during my work as a research assistant at the Institute for Materials Applications in Mechanical Engineering (IWM). This work would have never been accomplished without the help and support of many people, to whom I am deeply indebted and would like to express my deepest gratitude.

Firstly, I am very grateful to Univ.-Prof. Dr.-Ing. Christoph Broeckmann for giving me the opportunity to work at the Institute. I am thankful to him for the supervision, support, and encouragement. I would like to also thank Apl.-Prof. Dr. Martin Bram for not only accepting to evaluate my work but also for the very good collaboration during the joint research activities and numerous discussions. I would like to thank Prof. Dr.-Ing. K. Bobzin for accepting to chair the examination committee.

I would like to express my gratitude to Dr.-Ing. Anke Kaletsch for her constant professional support over the years at the institute and for her guidance. Her office door was always open for any discussion. I would also like to thank her for the correction of my dissertation and publication manuscripts. I am also thankful to M.Sc. Yuanbin Deng for supporting me as a group leader of Process Simulation group which I was a part of, and my sincere thanks go to M.Sc. Simone Herzog and M.Sc. Sofia Fries for their support.

The funding by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) under the Priority Programme (SPP) 1959/1 is highly acknowledged. I also take this opportunity to thank the members of the Priority Programme for the various discussions at the yearly colloquiums.

I would like to thank all colleagues at IWM with whom I have worked with. My special thanks go to Dipl.-Ing. Alexander Bezold for providing me with the opportunity to work in the institute as a Master Thesis student. I would like to thank all my office colleagues in the FEM office for the professional support and the very pleasant working atmosphere: M.Sc. Stanley van Kempen, M.Sc. C. Van Nguyen, M.Sc. Ali Rajaei, M.Sc. Geng Chen, M.Sc. Chao Liu, M.Sc. Manuel Wassenberg, and M.Sc. Siyuan Qin. I also thank B.Sc. Mathias Krämer and the labs group leader Vanessa Gebhardt for their support in the numerous experiments and investigations.

Lastly, I would also like to thank my parents and in-laws for their encouragement and belief in me. My heartfelt gratitude goes to my dear wife Manaswini Pisapaty for her endless support and understanding.

Aachen, July 2021

Sree Koundinya Sistla

Abstract

Field Assisted Sintering Technology, also known as Spark Plasma Sintering (FAST/SPS), is a new innovative sintering and synthesis technique. Due to the way electrical current flows through a tool system, high heating rates and short cooling periods are achieved. With assistance from the application of external pressure, it enables the drastic reduction of the sintering time, thereby reducing the production time and improving the microstructure and material properties. Finite Element Method (FEM) has been proven to be the best numerical tool to visualize the FAST/SPS process, and numerous modelling methods have been proposed in the literature. Although detailed modelling procedures are available, certain physical aspects have been neglected, such as the effect of electrical field/current on the microstructure evolution during sintering.

In this work, a Multiphysics FEM model has been developed to investigate the sintering of a broad spectrum of materials. Gadolinium doped ceria (GDC) has been chosen as oxide ceramic material due to its high electrochemical activity and stainless steel 316L (SS 316L) because of its simplicity in understanding the sintering mechanisms. The sintering kinetics were studied in detail, and sintering mechanisms for both materials have been proposed.

At elevated temperatures and dwell periods, the GDC samples exhibited the development of asymmetrical microstructures even under low electrical fields (< 5 V/cm). This effect has been observed for the first time. Enhanced grain growth was observed at the anode region, and this was attributed to the migration of oxygen ions under an electrical field to the anode, which led to high grain boundary mobility in that region. This experimental observation was also successfully modeled, with the simulations showing accurate results. Furthermore, the modelling procedure was also verified by using the other class of material, SS 316L. In this case, the effect of pressure on sintering was studied and numerically verified. Towards the end of the work, to validate the modelling procedure, simulations were carried out to simulate the sintering process of complex shaped geometries. The validation was realized by simulating the FAST/SPS of a SS 316L. The simulation was carried out by implementing a closed-loop control system (Proportional-Integral-Derivative (PID) controller) in the FEM simulation, which made the models independent of FAST/SPS experiments.

Zusammenfassung

Feldunterstützten Sintern/Spark Plasma Sintern (FAST/SPS) ist ein modernes innovatives Sinter- und Syntheseverfahren. Aufgrund der Art des elektrischen Stromflusses durch ein Werkzeugsystem, werden hohe Aufheizraten und kurze Abkühlzeiten realisiert. Die Anwendung von externem Druck ermöglicht die drastische Verkürzung der Sinterzeit, wodurch die Produktionsdauer verkürzt und die Materialeigenschaften verbessert werden. Die Finite-Elemente-Methode (FEM) hat sich als das beste numerische Werkzeug zur Visualisierung des FAST/SPS-Prozesses erwiesen, und in der Literatur wurden zahlreiche Modellierungsmethoden entwickelt. Obwohl detaillierte Modellierungsverfahren zur Verfügung stehen, werden bisher bestimmte physikalische Aspekte in der Simulation vernachlässigt. So wurde z.B. über den Einfluss des elektrischen Felds/Stroms auf die Mikrostrukturentwicklung während des Sinterns, nicht berichtet und diese nicht vollständig verstanden.

In dieser Arbeit wurde ein Multi Physics-FEM-Modell entwickelt, um das Sintern von unterschiedliche Werkstoffgruppen zu untersuchen. Gadolinium-dotiertes Ceroxid (GDC) wurde aufgrund seiner hohen elektrochemischen Aktivität als oxidkeramische Werkstoff und ein austenitischer korrosionsbeständiger Stahl 316L (SS 316L) als metallischer Werkstoff ausgewählt. Im Rahmen dieser Arbeit wurden FAST/SPS-Experimente durchgeführt und die gesinterten Proben charakterisiert, um Modelle zur Abbildung der Sinterkinetik (Verdichtung und Kornwachstum) zu ermitteln.

Nach bestem Wissen des Autors zeigten die GDC-Proben zum ersten Mal bei erhöhten Temperaturen und Haltezeiten die Entwicklung einer asymmetrischen Mikrostruktur auch bei geringen elektrischen Feldern (< 5 V/cm). Es wurde ein stärkeres Kornwachstum im Anodenbereich beobachtet, was auf die Migration von Sauerstoff-Ionen zur Anode unter einem elektrischen Feld zurückgeführt wurde. Diese experimentellen Beobachtungen wurden erfolgreich modelliert, wobei die Simulationen genaue Ergebnisse zeigten. Darüber hinaus wurde das Modellierungsverfahren auch unter Verwendung der anderen Werkstoffgruppe, SS 316L, verifiziert. In diesem Fall wurde die Auswirkung des Drucks auf das Sinterverhalten untersucht und numerisch verifiziert. Gegen Ende der Arbeit wurden zur Validierung des Modellierungsverfahrens Simulationen des Sinterprozesses von komplex geformten Geometrien durchgeführt. Dies wurde durch die Simulation des Sinterns eins Zahnrades aus SS 316L realisiert. Die Simulation wurde durch die Implementierung eines geschlossenen Regelkreises (Proportional-Integral-Derivativ-Regler) in die FEM-Simulation durchgeführt, wodurch die Modelle unabhängig von FAST/SPS-Experimenten wurden.

Table of Contents

Table of ContentsI						
Li	st of Fig	gures	V			
Li	st of Ta	bles	KI			
Sy	Symbols and AbbreviationsXIII					
1	Intro	oduction	1			
2	Fun	damentals and state of the art	5			
	2.1 5	Sintering	5			
	2.1.1	Stages of sintering	6			
	2.1.2	2 Transport mechanisms during sintering	7			
	2.1.3	Pressure-assisted sintering	8			
	2.1.4	Novel sintering techniques under electromagnetic fields	9			
	2.2 F	ield assisted sintering technology / spark plasma sintering (FAST/SPS)1	1			
	2.2.1	I Technology1	1			
	2.2.2	2 Effect of high heating rates1	2			
	2.2.3	B Effect of pressure 1	3			
	2.2.4	Effect of the electrical field1	4			
	2.2.5	5 Numerical simulation of FAST/SPS1	7			
	2.2.6	Proportional-Integral-Derivative (PID) controller2	21			
	2.3 N	Aaterials under investigation2	2			
	2.3.1	Gadolinium doped ceria (GDC)2	22			
	2.3.2	2 Austenitic stainless steel (SS 316L)2	23			
	2.4 8	Summary of the state of the art2	4			
3	Moti	vation and Objective2	27			
4	Exp	erimental and numerical methods2	29			
	4.1 F	AST/SPS experiments2	9			
	4.2 0	Characterization methods3	0			

Ш		Table of Contents	
	4.2.1	Microstructure characterization	31
	4.2.2	Thermophysical characterization	
2	1.3 Nu	umerical methods	32
	4.3.1	A coupled thermal electrical model	
	4.3.2	Constitutive model	
	4.3.3	Viscoplastic implementation in ABAQUS	
	4.3.4	PID controller implementation in ABAQUS	
5	Expe	rimental investigations and results	41
Ę	5.1 Gi	raphite	41
Ę	5.2 Ga	adolinium doped ceria (GDC)	42
	5.2.1	GDC microstructure characterization	43
	5.2.2	GDC thermophysical characterization	43
	5.2.3	Densification kinetics	
	5.2.4	Grain growth kinetics	47
	5.2.5	Effect of the electrical field	
	5.2.6	Intermediate discussion and summary 1	
Ę	5.3 St	ainless steel 316L (SS 316L)	61
	5.3.1	SS 316L microstructure characterization	62
	5.3.2	SS 316L thermophysical characterization	64
	5.3.3	Densification kinetics	64
	5.3.4	Grain growth kinetics	67
	5.3.5	Intermediate discussion and summary 2	69
6	Nume	rical results and discussion	71
6	6.1 Ca	alibration of contact conductivities	73
	6.1.1	Verification of simulation results of tool assembly A1	73
	6.1.2	Verification of simulation results of tool assembly A2	75
6	6.2 M	odelling of GDC	

			Table of Contents	
	6.2	2.1	Verification of temperature distribution	77
	6.2	2.2	Verification of low-temperature densification and grain growth	79
	6.2	2.3	Effect of electrical field on microstructure evolution and discussion	82
	6.2	2.4	Intermediate discussion and summary 3	90
6	6.3	Mo	odelling of Stainless steel 316L (SS 316L)	92
	6.3	3.1	Verification of temperature distribution	92
	6.3	3.2	Results of densification and grain growth and discussion	96
	6.3	3.3	Simulation of complex shapes	101
	6.3	3.4	Intermediate discussion and summary 4	107
7	Su	ımm	nary and outlook	109
7	7.1	Su	mmary	109
7	7.2	Οι	itlook	111
8	Re	efere	ences	113
9	At	tacł	1ments	131
ç	9.1	Sa	mple preparation plan for grinding and polishing	131
ç	9.2	Im	age analysis steps using ImageJ	132
ç	9.3	Gr	aphite	133
	9.3	3.1	Thermophysical Properties of Graphite	133
ç	9.4	GE	DC	135
	9.4	1.1	Grain Sizes of sintered samples	135
	9.4	1.2	SEM images of GDC samples	137
	9.4	1.3	Relative density of GDC sintered samples	139
	9.4	1.4	Thermophysical Properties of GDC	140
ç	9.5	SS	316L	141
	9.5	5.1	Relative density of SS 316 sintered samples	141
	9.5	5.2	Grain Sizes of sintered samples	142
	9.5	5.3	Thermophysical Properties of SS 316L	143

IV	Table of Contents
9.6	PID controller flow chart145