VAPOUR PHASE GROWTH AND NANOMECHANICAL BEHAVIOUR OF VO_2 MICROCRYSTALS ACROSS PHASE TRANSITION

A THESIS

submitted by

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of

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Preface

The present Ph.D. thesis is submitted in candidacy for a Ph.D. degree from Indian Institute of Technology, Mandi (IIT). The work presented in this thesis was carried out during the period of August 2014 to September 2018 at Nanoscale and Device laboratory at School of Engineering under supervision of Dr. Viswanath Balakrishnan.

The project was fully funded by Ministry of Human Resource Development (MHRD), Government of India and Indian Institute of Technology, Mandi (IIT). The main goal of the project was to investigate the mechanical behaviour and actuation in phase change materials at small scale for enabling applications in cantilever, MEMS applications.

IIT Mandi, established in 2009, has rapidly risen among the premier institutes of India. It is located in Kamand valley on the banks of Uhl, a tributary of the river Beas. Kamand is approximately 14 kms from Mandi town and has an average elevation of 1044 meters from sea level. There is great variation in the climatic conditions of Himachal due to extreme variation in elevation. The climate varies from hot and sub-humid tropical in the southern tracts to cold, alpine and glacial in the northern and eastern mountain ranges with more elevation.

To Punit, who saved me from

my monochromatic nihilism and my pathological altruism.

"I am and always will be the optimist.

The hoper of far-flung hopes and the dreamer of improbable dreams."

— Doctor Who

Declaration

I hereby declare that the entire work embodied in this Thesis is the result of investigations carried out by me in the ***School of Engineering,*** Indian Institute of Technology Mandi, under the supervision of ***Dr. Viswanath Balakrishnan,*** and that it has not been submitted elsewhere for any degree or diploma. In keeping with the general practice, due acknowledgements have been made wherever the work described is based on finding of other investigators.

Place: Signature:

Date: Name: Davinder Singh (D14014)

Certificate

This is to certify that the thesis entitled "**Vapour Phase Growth and Nanomechanical Behaviour of VO_2 Microcrystals across Phase Transition**" submitted by Davinder Singh, a Research Scholar in the School of Engineering, Indian Institute of Technology Mandi, for the award of the degree of **DOCTOR OF PHILOSOPHY**, is a record of an original research work carried out by him under my supervision and guidance. The thesis has fulfilled all the requirements as per the regulations of the Institute. The results embodied in this thesis have not been submitted to any other University or Institute for the award of any degree or diploma.

Signature:

Dr. Viswanath Balakrishnan

Date:

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List of Publications

1. D. Singh and B. Viswanath., Direct Measurement of Nanomechanical Actuation Across Phase Transition in VO_2 Crystals, Scr. Mater., (2017).
2. D. Singh and B. Viswanath, In situ Nanomechanical Behaviour of Coexisting Insulating and Metallic Domains in VO_2 Microbeams, J. Mater. Sci., (2017).
3. D. Singh, C.S. Yadav, and B. Viswanath, Magnetoresistance Across Metal–Insulator Transition in VO_2 Micro Crystals, Mater. Lett., (2017).
4. D. Singh and B. Viswanath, In situ Thermo-Mechanical Bending Behaviour of VO_2 Microcantilevers Across the Phase Transition, Journal of Micromechanics and Microengineering, (2018).
5. D. Singh, Prashant Mittal, Nitya Nand Gosvami and B. Viswanath, *Switchable Friction Across Insulator-Metal Transition in* VO_2, to be submitted
6. D. Singh, Piyush Avasthi, B. Viswanath, CVD Growth and Properties of VO_2 Platelets on Quartz Substrate for IR Modulation Applications, Under Preparation
7. D. Singh and B. Viswanath, Measurement of Fracture Toughness Across Insulator-Metal Transition in VO_2 Microcrystals, Under Preparation

Abstract

Phase change materials are playing an increasingly important role in small scale mechanical devices. My dissertation addressed the question of how temperature affects the mechanical behaviour of Vanadium Dioxide across the structural transition. I have also formulated various nanoindentation testing techniques to study the mechanical behaviour of various microscale structures. Considering the multitude of application of VO_2 in micro electromechanical devices, the immediate objective of this research project was to measure the elastic modulus and hardness of VO_2 across the phase transition. Brief findings of consistent work done over last four years is given as follows:

- Coexisting phases in VO_2 are identified using Raman, optical imaging and electrical measurements. Site specific *in situ* nanoindentation confirm the abrupt increase in elastic modulus (~ 17 GPa) and nanohardness (1 GPa) across the transition from monoclinic (insulator) to rutile (metal) phase.
- Electron back scattered diffraction (EBSD) analysis has been used to confirm the [100] orientation of monoclinic VO_2 crystals selected for nanomechanical actuation measurement. The measured spontaneous strain across phase transition is found to be reversible and demonstrates that VO_2 micro crystals can be used to make highly efficient thermal actuators.
- Bending behaviour study of VO_2 microcantilever show higher stiffness for metallic phase of VO_2. Spring constant of the microcantilever was found to be increased by a factor of 1.3 during the structural phase transition of VO_2 from monoclinic to rutile structures.
- Growth of VO_2 platelets has been successfully achieved for IR camouflage applications.
- Switchable friction behaviour has been demonstrated for two phases of VO_2 and may have applications in gripping devices.
- Fracture toughness measurement is done using nanoindentation tests and high toughness value for metallic phase of VO_2 is observed as compared to insulating phase.

Overall experiments on single crystalline VO_2 led to a mechanistic understanding of mechanical behaviour of VO_2 suitable for micro-actuators, cantilevers, grasping and climbing robotic systems and IR modulation devices.

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