

Thin Film Shape Memory Alloys: Fundamentals and Device Applications

The first dedicated to this exciting and rapidly growing field, this book enables readers to understand and prepare high-quality, high-performance TiNi shape memory alloys (SMAs). It covers the properties, preparation, and characterization of TiNi SMA thin films with particular focus on the latest technologies and applications in MEMS and biological devices. Basic techniques and theory are covered to introduce new-comers to the subject, whilst various sub-topics, such as film deposition, characterization, post treatment, and applying thin films to practical situations, appeal to more informed readers. Each chapter is written by expert authors, providing an overview of each topic and summarizing all the latest developments, making this an ideal reference for practitioners and researchers alike.

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Thin Film Shape Memory Alloys

Fundamentals and Device Applications

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Preface

Shape memory alloys (SMAs) are materials that, after being severely deformed, can return to their original shape upon heating. These materials possess a number of desirable properties, namely, high power to weight (or force to volume) ratio, thus the ability to induce large transformation stress and strain upon heating/cooling, pseudoelasticity (or superelasticity), high damping capacity, good chemical resistance and biocompatibility, etc. These unique features have attracted much attention to the potential applications of SMAs as smart (or intelligent) and functional materials. More recently, thin film SMAs have been recognized as a new type of promising and high-performance material for microelectromechanical system (MEMS) and biological applications.

Among these SMA films, TiNi based films are the most promising ones. They are typically prepared by a sputtering method. Other technologies, e.g., laser ablation, ion beam deposition, arc plasma ion plating, plasma spray and flash evaporation, have also been reported in the literature, but with some intrinsic problems. It is well known that the transformation temperatures, shape memory behaviors and superelasticity of the sputtered TiNi films are sensitive to metallurgical factors (alloy composition, contamination, thermomechanical treatment, annealing and aging processes, etc.), sputtering conditions (co-sputtering with multi-targets, target power, gas pressure, target-to-substrate distance, deposition temperature, substrate bias, etc.), and the application conditions (loading conditions, ambient temperature and environment, heat dissipation, heating/cooling rate, strain rate, etc.).

The main advantages for MEMS applications of TiNi thin film include high power density, large displacement and actuation force, low operation voltage, etc. The work output per unit volume of thin film SMA exceeds that of all other microactuation materials and mechanisms. Application of SMA films in MEMS also facilitates the simplification of mechanisms with flexibility in design and creation of clean, friction free and non-vibrating movement. The phase transformation in SMA thin films is accompanied by significant changes in the mechanical, physical, chemical, electrical and optical properties, such as yield stress, elastic modulus, hardness, damping, shape recovery, electrical resistivity, thermal conductivity, thermal expansion coefficient, surface roughness, vapor permeability and dielectric constant, etc. TiNi thin films are sensitive to environmental changes

such as thermal, stress, magnetic or electrical fields, and thus should be ideal for applications in microsensors as well.

Since TiNi films can provide a large force and/or large displacement in actuation, most applications of TiNi films in MEMS are focused on microactuators. Micro-pumps, microvalves, microgrippers, springs, microspacers, micropositioners and microrappers are typical among others that have been realized. TiNi based micro-pumps and microvalves are attractive for many applications, for instance, implantable drug delivery, chemical analysis and analytical instruments, etc. Grasping and manipulating small or micro-objects with high accuracy is required for a wide range of important applications, such as microassembly in microsystems, endoscopes for microsurgery, and drug injection micromanipulators for cells.

The good biocompatibility of TiNi films is promising for their biological applications, which is a huge market at present and is still growing. At present, increasing attention has been paid to the use of TiNi thin film for minimally invasive surgery, microstents and bioMEMS applications. Microactuators made of TiNi thin films may be used to infuse drugs, or be placed in strategic locations in the body to assist circulation. TiNi SMA thin films, in the superelastic state, are also promising as compliant elements in MEMS and biological devices.

The development of TiNi based SMA thin films and their microactuators has achieved considerable progress in recent years. This was largely driven by a fast expansion of, in particular, MEMS and biological communities, in which the demand for novel actuators and biological applications has been growing dramatically. As such, a timely review of the important issues pertaining to the preparation of high quality and high performance shape memory TiNi thin films and the technical applications of these films is necessary. This book aims to serve this purpose. We believe that this is the first book dedicated to thin film TiNi SMAs; it covers not only the state-of-the-art technologies for thin film SMAs (preparation and characterization), but also their applications, in particular, in MEMS and biomedical devices. This book should naturally serve not only as an introduction to those who want to know more about this exciting field, but also as a technical handbook for those who have some knowledge but want to know more. Hence, it is an essential reference book both for a better understanding of the fundamental issues in technical aspects and for catching up with the current developments in technologies and applications in new frontiers.

The book is naturally divided into two parts, namely, technologies (from Chapter 1 to Chapter 9) and applications (Chapter 10 to Chapter 19). The first part is focused on the fundamental issues of sputter-deposited TiNi based SMA thin films, covering a general overview (Chapter 1); the basics of martensitic transformation (Chapter 2); deposition technologies (Chapter 3), Ti/Ni multi-layer thin films (Chapter 4); microstructure, crystallization, mechanical properties and stress evolution in thin film SMA (Chapters 5 to 7); as well as advanced post-treatment of thin film SMA including ion implantation and laser annealing (Chapters 8 and 9). The second part is devoted to the device applications based on TiNi based SMA thin film, focusing mainly on MEMS and biological applications.

It covers: an overview of applications (Chapter 10); theory and simulation of shape memory microactuators (Chapter 11); MEMS devices of microvalves, micro-pumps, microcages, micromirrors, superelastic thin film for medical applications, and thin film composite microactuators (Chapters 12 to 19).

The editors express their heartfelt thanks to the distinguished international team of contributors whose scientific efforts unite to form this book. We also express special thanks to the staff, Dr. Michelle Carey and Miss Sarah Matthews, at Cambridge University Press for their assistance.

Abstracts of chapters

1 Overview of sputter-deposited TiNi based thin films

S. MIYAZAKI, Y. Q. FU AND W. M. HUANG

Abstract: The motivation for fabricating sputter-deposited TiNi base shape memory alloy (SMA) thin films originates from the great demand for the development of powerful microactuators, because actuation output (force and displacement) per unit volume of thin film SMA exceeds those of other microactuation mechanisms. Stable shape memory effect and superelasticity, which are equivalent to those of bulk alloys, have been achieved in sputter-deposited TiNi thin films. Narrow transformation temperature hysteresis and high transformation temperatures were also achieved in TiNiCu and TiNi(Pd or Hf) thin films, respectively. In the meantime, unique microstructures consisting of non-equilibrium compositions and nanoscale precipitates in the matrix have been found in Ti-rich TiNi thin films that were fabricated from an amorphous condition by annealing at a very low temperature. Several micro-machining processes have been proposed to fabricate the prototypes of microactuators utilizing TiNi thin films. This chapter will review the recent development of the above-mentioned topics relating to sputter-deposited TiNi based thin films. Some critical issues and problems in the development of TiNi thin films are discussed, including preparation and characterization considerations, residual stress and adhesion, frequency improvement, fatigue and stability, and thermomechanical modeling. Recent developments in the microdevices based on SMA thin films are also summarized.

2 Martensitic transformation in Ti-Ni alloys

S. MIYAZAKI

Abstract: The basic characteristics of the martensitic transformation of Ti-Ni shape memory alloys are described. They include the crystal structures of the parent and martensite phases, the recoverable strain associated with the martensitic transformation, the transformation temperatures, the temperature and orientation

dependence of deformation behavior, etc. Shape memory and superelasticity related to the martensitic transformation are also explained.

3 Deposition techniques for TiNi thin film

A. D. JOHNSON

Abstract: Direct current vacuum sputter deposition is the commonly used method of creating TiNi thin film. Polished silicon wafers are a preferred substrate. The limitations on composition and impurities are similar to those for bulk material. These limitations impose severe constraints on sputtering conditions for obtaining optimal performance of the resulting material. Obtaining material with desirable shape memory properties, uniform composition, and uniform thickness requires an understanding and control of the processes used. With sputter deposition it is possible to produce thin films with a range of transition temperatures from 173 K to 373 K. Superelastic thin film can be made without cold work. After deposition, photolithography and chemical etching are used to create shapes and combine thin film with other materials to produce microdevices. Producing thin film with shape memory properties is not difficult, but to obtain uniformity and high yield requires specialized equipment and great care in process control. This chapter introduces some specific recommendations for fabrication of TiNi thin film and its incorporation in useful devices. Applications of TiNi thin film are described elsewhere (see Chapter 10).

4 TiNi multilayer thin films

H. CHO AND S. MIYAZAKI

Abstract: The martensitic transformation temperatures of TiNi shape memory alloys are strongly affected by composition. However, the composition of the TiNi thin film fabricated by a conventional sputtering method using a TiNi alloy target is not easy to adjust precisely. In order to control the composition of thin films, a dual-source sputtering method using pure Ti and Ni targets can be an alternative candidate for fabricating TiNi multilayer thin films, which can be converted to TiNi alloy thin films by heat-treatment for alloying. This chapter explains the fabrication method and alloying process of the TiNi multilayer thin films in addition to the shape memory properties of the TiNi thin films made by alloying the TiNi multilayer thin films.

5 Crystallization and microstructural development

A. G. RAMIREZ, X. HUANG AND H.-J. LEE

Abstract: This chapter focuses on the crystallization of TiNi shape memory alloy thin films. These materials are commonly sputter-deposited in an amorphous

form and require high-temperature thermal treatments to create their crystalline (actuating) form. The microstructures that emerge during crystallization depend on the nucleation and growth kinetics. This chapter briefly surveys crystallization theory and methods to determine these kinetic parameters such as calorimetry, X-ray diffraction and microscopy. Novel microscopy methods have also been developed to provide a robust description that can give rise to the prediction of microstructures. In addition to presenting these tools, this chapter will also survey various factors that influence crystallization and microstructural development, which include annealing temperature, composition, substrate materials and film thickness.

6 Mechanical properties of TiNi thin films

A. ISHIDA

Abstract: This chapter is devoted to the mechanical properties of TiNi thin films. In this chapter, the shape memory behavior, two-way shape memory effect, superelasticity, and stress–strain curves of sputter-deposited TiNi films are discussed with special attention to unique microstructures in Ti-rich TiNi films.

7 Stress and surface morphology evolution

Y. Q. FU, W. M. HUANG, M. CAI AND S. ZHANG

Abstract: The residual stress in a film is an important topic of extensive studies as it may cause film cracking and peeling off, or result in deformation of the MEMS structure, deterioration of the shape memory and superelasticity effect. In this chapter, the stress in thin film shape memory alloys is characterized. The recovery stress, stress rate and stress–strain relationship during phase transformation are introduced. The dominant factors affecting stress evolution during deposition, post-annealing and phase transformation are discussed. The mechanisms for the stress-induced surface relief, wrinkling and reversible trench have also been studied. New methods for characterization of stress-induced surface morphology changes have been introduced, including atomic force microscopy (AFM) and photoemission electron microscopy (PEEM).

8 Ion implantation processing and associated irradiation effects

T. LAGRANGE AND R. GOTTHARDT

Abstract: In this chapter, we describe the influence of ion implantation on the microstructural modifications in TiNi SMA thin films. We focus on investigations involving 5 MeV Ni ion irradiation since it can be used as a means to selectively

alter the transformation characteristics and to develop NiTi based thin film actuator material for MEMS devices. The primary effects of ion implantation on microstructure are summarized.

9 Laser post-annealing and theory

W. M. HUANG AND M.H. HONG

Abstract: TiNi shape memory thin films have great potential as an effective actuation material for micro-sized actuators. However, a high temperature (above approximately 723 K) is required in order to obtain crystalline thin films either during deposition (e.g., sputtering) or in post-annealing. Such a high temperature is not fully compatible with the traditional integrated circuit techniques, and thus brings additional constraint to the fabrication process. Laser annealing provides an ideal solution to this problem, as the high temperature zone can be confined well within a desired small area at a micrometer scale. In this chapter, we demonstrate the feasibility of local laser annealing for crystallization in as-deposited amorphous TiNi thin films and present a systematic study of the theories behind this technique.

10 Overview of thin film shape memory alloy applications

A. D. JOHNSON

Abstract: This chapter discusses properties affecting thin film applications and resulting devices that have been developed since sputtered TiNi thin film with shape memory properties was first demonstrated in 1989. As the shape memory alloy technology has matured, the material has gradually gained acceptance. TiNi thin film shape memory alloy (SMA) exhibits intrinsic characteristics similar to bulk nitinol: large stress and strain, long fatigue life, biocompatibility, high resistance to chemical corrosion, and electrical properties that are well matched to joule heating applications. In addition, thin film dissipates heat rapidly so that it can be thermally cycled in milliseconds. These properties make TiNi thin film useful in making microactuators. Micro electro mechanical (MEMS) processes – specifically photolithography, chemical etching, and the use of sacrificial layers to fabricate complex microstructures – combine TiNi thin film with silicon to provide a versatile platform for fabrication of micro-devices. A variety of microdevices has been developed in several laboratories, including valves, pumps, optical and electrical switches, and intravascular devices. Interest in thin film applications is increasing as evidenced by the number of recent publications and patents issued. Intravascular medical devices are currently in clinical trials. The future for thin film devices, especially in medical devices, seems assured despite the fact that to this day no “killer application” has emerged.

11 Theory of SMA thin films for microactuators and micropumps

Y. C. SHU

Abstract: This chapter summarizes several recent theoretical and computational approaches for understanding the behavior of shape memory films from the microstructure to the overall ability for shape recovery. A new framework for visualizing microstructure is presented. Recoverable strains in both single crystal and polycrystalline films are predicted and compared with experiments. Some opportunities for new devices and improvements in existing ones are also pointed out here.

12 Binary and ternary alloy film diaphragm microactuators

S. MIYAZAKI, M. TOMOZAWA AND H.Y. KIM

Abstract: TiNi based shape memory alloy (SMA) thin films including TiNi, TiNiPd and TiNiCu have been used to develop diaphragm microactuators. The TiNi film is a standard material and the ternary TiNi. The TiNiPd alloy films have their own attractive characteristics when compared with the TiNi films. The TiNiPd alloy is characterized by high transformation temperatures so that it is expected to show quick response due to a higher cooling rate: the cooling rate increases with increasing the temperature difference between the transformation temperature and room temperature, which is the minimum temperature in conventional circumstances. The martensitic transformation of the TiNiCu and the R-phase transformation of the TiNi are characterized by narrow transformation hystereses which are one-fourth and one-tenth of the hysteresis of the martensitic transformation in the TiNi film. Thus, these transformations with a narrow hysteresis are also attractive for high response microactuators. The working frequencies of two types of microactuators utilizing the TiNiPd thin film and the TiNiCu film reached 100 Hz while the working frequency of the microactuator using the R-phase transformation reached 125 Hz.

13 TiNi thin film devices

K. P. MOHANCHANDRA AND G. P. CARMAN

Abstract: This chapter provides a brief review of TiNi thin film devices, both mechanical and biomedical, that have been studied during the last decade. Prior to reviewing the devices, we first provide a description of the physical features that are critical in these devices, including deposition, residual stresses and fabrication. In general, this chapter concludes that the main obstacle for implementing devices today remains the control of TiNi properties during manufacturing. If this can be overcome, the next issue is to develop acceptable

micromachining techniques. While several techniques have been studied, considerable work remains on fully developing processes that would be required in mass manufacturing processes. Finally, if these issues can be resolved, the area with the most promise is biomedical devices, the argument here being that biomedicine remains one of the major applications areas for macroscopic TiNi structures today.

14 Shape memory microvalves

M. KOHL

Abstract: Microvalves are a promising field of application for shape memory alloy (SMA) microactuators as they require a large force and stroke in a restricted space. The performance of SMA-actuated microvalves does not only depend on SMA material properties, but also requires a mechanically and thermally optimized design as well as a batch fabrication technology that is compatible with existing microsystems technologies. The following chapter gives an overview of the different engineering aspects of SMA microvalves and describes the ongoing progress in related fields. Different valve types based on various design–material–technology combinations are highlighted. The examples also demonstrate the opportunities for emerging new applications.

15 Superelastic thin films and applications for medical devices

C. ZAMPONI, R. L. DE MIRANDA AND E. QUANDT

Abstract: Superelastic shape memory materials are of special interest in medical applications due to the large obtainable strains, the constant stress level and their biocompatibility. TiNi sputtered tubes have a high potential for application as vascular implants, e.g. stents, whereas superelastic TiNi-polymer-composites could be used for novel applications in orthodontics and medical instrumentation as well as in certain areas of mechanical engineering. In orthodontic applications, lowering the forces that are applied to the teeth during archwire treatment is of special importance due to tooth root resorption, caused by the application of oversized forces. Furthermore, the use of superelastic materials or composites enables the application of constant forces independent of diminutive tooth movements during the therapy due to the superelastic plateau. Superelastic TiNi thin films have been fabricated by magnetron sputtering using extremely pure cast melted targets. Special heat treatments were performed for the adjustment of the superelastic properties and the transformation temperatures. A superelastic strain exceeding 6% at 36°C was obtained.

16 Fabrication and characterization of sputter-deposited TiNi superelastic microtubes

P. J. BUENCONSEJO, H.-Y. KIM AND S. MIYAZAKI

Abstract: A novel method of fabricating TiNi superelastic microtubes with a dimension of less than 100 μm is presented in this chapter. The method was carried out by sputter-deposition of TiNi on a Cu-wire substrate, and after deposition the Cu wire was removed by etching to produce a tube hole. The shape-memory/superelastic behavior and fracture strength of the microtubes were characterized. The factors affecting the properties and a method to produce high-strength superelastic TiNi microtubes are discussed.

17 Thin film shape memory microcage for biological applications

Y. Q. FU, J. K. LUO, S. E. ONG AND S. ZHANG

Abstract: This chapter focuses on the fabrication and characterization of microcage for biopsy applications. A microcage based on a free standing film could be opened/closed through substrate heating with a maximum temperature of 90 °C, or Joule heating with a power less than 5 mW and a maximum response frequency of 300 Hz. A TiNi/diamond-like carbon (DLC) microcage has been designed, analyzed, fabricated and characterized. The bimorph structure is composed of a top layer of TiNi film and a bottom layer of highly compressively stressed DLC for upward bending once released from the substrate. The fingers of the microcage quickly close because of the shape memory effect once the temperature reaches the austenite transformation point to execute the gripping action. Opening of the microcage is realized by either decreasing the temperature to make use of the martensitic transformation or further increasing the temperature to use the bimorph thermal effect. The biocompatibility of both the TiNi and DLC films has been investigated using a cell-culture method.

18 Shape memory thin film composite microactuators

E. QUANDT

Abstract: Shape memory thin film composites consisting of at least one shape memory thin film component are of special interest as microactuators since they provide two-way shape memory behavior without any training of the shape memory material. Furthermore, they allow the realization of novel concepts like bistable or phase-coupled shape memory actuators. The potential of shape memory thin film composite microactuators is discussed in view of possible applications.

19 TiNi thin film shape memory alloys for optical sensing applications

Y. Q. FU, W. M. HUANG AND C. Y. CHUNG

Abstract: This chapter focuses on the optical sensing applications based on TiNi films. When the TiNi film undergoes a phase transformation, both its surface roughness and reflection change; this can be used for a light valve or on–off optical switch. Different types of micromirror structures based on sputtered TiNi based films have been designed and fabricated for optical sensing applications. Based on the intrinsic two-way shape memory effect of free standing TiNi film, TiNi cantilever and membrane based mirror structures have been fabricated. Using bulk micromachining, TiNi/Si and TiNi/Si₃N₄ bimorph mirror structures were fabricated. As one application example, TiNi cantilevers have been used for infrared (IR) radiation detection. Upon absorption of IR radiation, TiNi cantilever arrays were heated up, leading to reverse R-phase transition and bending of the micro-mirrors.