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Title

Abstract

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Micro and Macro Deformation of Single Crystal NiTi

We present experimental results on the instrumented Vickers micro-indentation and compression of solutionized Ni-rich NiTi single crystals. The tests are conducted at room temperature where the solutionized Ti-50.9 at percent Ni material is 18 degrees above A_f and the solutionized Ti-51.5 at percent Ni material is more than 100 degrees above A_f. Aside from elastic deformation, it is discovered that dislocation motion and a reversible stress-induced martensitic transformation influence the micro-indentation response of Ti-50.9 at percent Ni, while the micro-indentation of Ti-51.5 at percent Ni only induces irreversible dislocation motion. The effect of the surface normal orientation on material hardness was negligible in the Ti-51.5 at percent Ni and followed trends anticipated by the activation of favorable slip systems in the Ti-50.9 at percent Ni. Compression tests on the identical Ti-50.9 at percent Ni samples revealed deformation by coupled stress-induced martensite and plastic flow, depending on the crystallographic orientation. The trends in hardness with surface normal orientation were not commensurate with the orientation dependence of the uniaxial compressive transformation or "yield" strength. The ramifications of the results in terms of comparing micro-indentation and macro-compression and the interactions between plasticity and the stress-induced martensitic transformation are discussed. [DOI: 10.1115/1.1416684]

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Abstract

CRITICAL REVIEW

Critical Review: Adhesion in surface micromechanical structures

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(Received 15 November 1996; accepted 15 November 1996)

We present a review on the state of knowledge of surface phenomena behind adhesion in surface micromechanical structures. After introducing the problem of release-related and in-use adhesion, a theoretical framework for understanding the various surface forces that cause strong adhesion of micromechanical structures is presented. Various approaches are described for reducing the work of adhesion. These include surface roughening and chemical modification of polycrystalline silicon surfaces. The constraints that fabrication processes such as release, drying, assembly, and packaging place on surface treatments are described in general. Finally, we briefly outline some of the important scientific and technological issues in adhesion and friction phenomena in micromechanical structures that remain to be clarified. © *1997 American Vacuum Society.*
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I. INTRODUCTION

Surface micromachining is defined as the fabrication of micromechanical structures from deposited thin films. Although the basic idea dates back to the 1960s,[1] it has been the focus of rapidly growing research and, recently, commercial applications.[2] Surface micromachining is one of the core technologies underlying microelectromechanical systems (MEMS), which promises to extend the benefits of microelectronic fabrication technology to sensing and actuating [illegible]e reason that surface micromachining has rap[illegible]d is the mature infrastructure for depositing, pat[illegible]etching thin films borrowed from silicon inte[illegible] technology. Another reason is the potential of [illegible]icrosystems, which incorporate surface micro[illegible]nsors or actuators together with integrated elec[illegible]he same substrate.[3] Early applications of this technology include the digital mirror display, which has on the order of 10^6 aluminum thin-film mirrors fabricated on top of a complementary metal–oxide–semiconductor (CMOS) static random access memory (SRAM) integrated circuit.[4,5] Integrated accelerometers for airbag deployment[6] and, recently, for more demanding applications,[7] use a suspended polycrystalline silicon (polysilicon) inertial sense element. BiCMOS electronics adjacent to the polysilicon microstructure sense its position capacitively and are used to apply balancing electrostatic forces. Vibratory rate gyroscopes[3] and other inertial sensors have been demonstrated in the same technology, making it feasible to have a six degree-of-freedom inertial measurement on a single silicon chip. Recently, a modular process that embeds the polysilicon microstructure in the wafer prior to CMOS fabrication was developed.[8]

Surface microstructures typically range from 0.1 to several μm in thickness with lateral dimensions of 10–500 μm, and lateral and vertical gaps to other structures or to the substrate of around 1 μm. A representative polysilicon-based integrated MEMS device[9] is shown in Fig. 1. The large surface area and small offset from adjacent surfaces makes these microstructures especially vulnerable to adhesion upon contact. As an example, the "pull-off" force of a displaced surface microstructure in contact with an adjacent surface ranges from a few μN for an airbag accelerometer sense element to nN for highly compliant microstructures with submicron flexure widths.[10] These forces are considerably weaker than interfacial forces, and hence, permanent adhesion results upon contact. Since in many microdevices, one is not only dealing with a vertical pull-off force but also with a peeling (friction) phenomenon, this problem is more generally called *stiction*, a term borrowed from the magnetic recording media industry.[11] The adhesion of the microstructure to adjacent surfaces can occur either during the final steps of the micromachining process (release-related adhesion) or after packaging of the device, due to overrange input signals or electromechanical instability (in-use adhesion).[12] The distinction between the two classes will be useful, since their causes and strategies for eliminating or minimizing adhesion differ.

It must be noted that high-aspect ratio microstructures of single crystal silicon[13,14] and other materials[15] are also susceptible to adhesion. These structures are typically sus-

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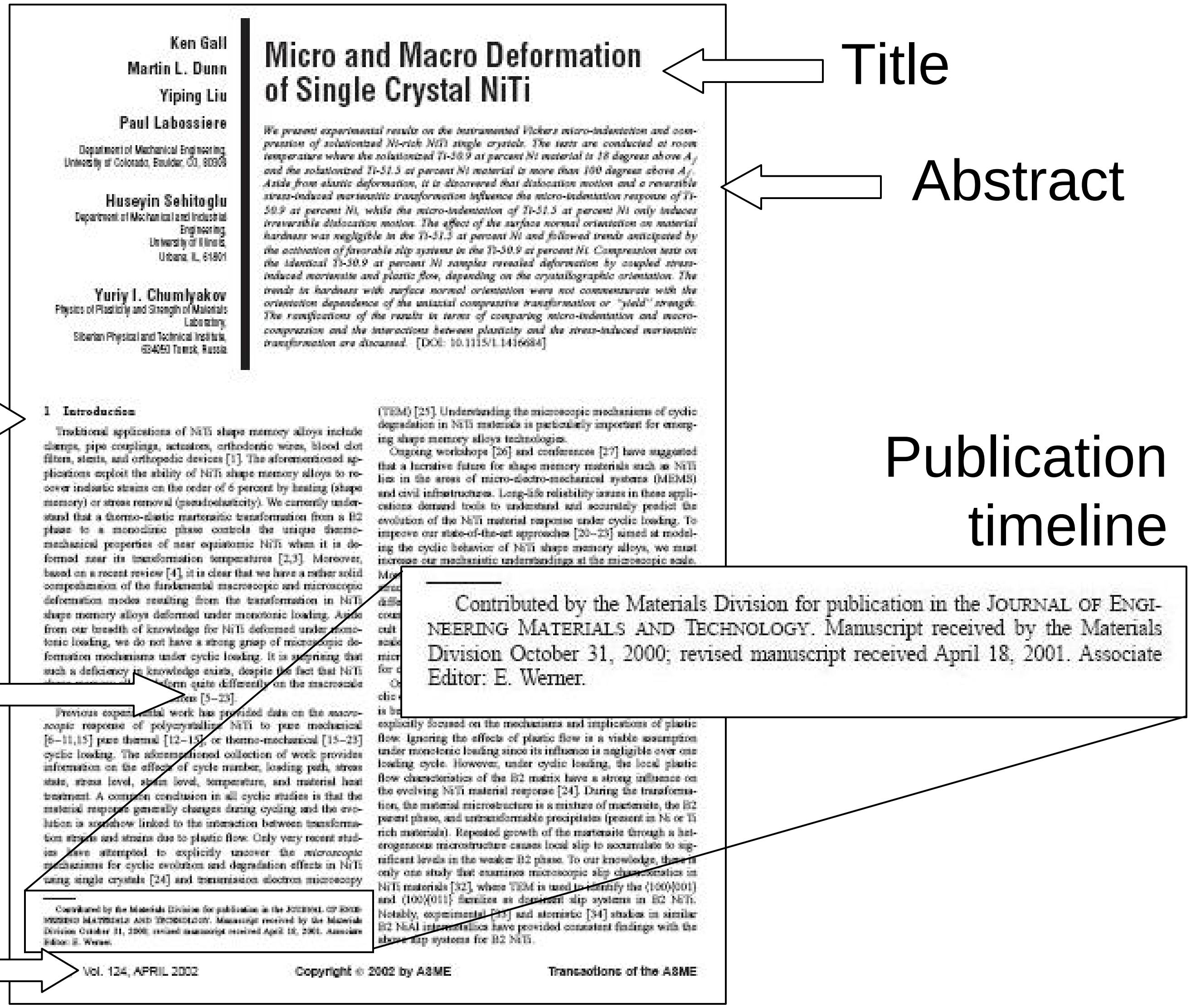

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5 Conclusions

1 Micro-indentation hardnesses as a function of surface normal orientation do not correlate with the macroscopic compressive transformation or "yield" strength. The indentation response of Ti-50.9 at percent Ni deformed 18 deg above A_f results in elastic and plastic deformation, and a stress-induced martensitic transformation. The material hardness with orientation correlates best with the resistance to dislocation motion. The hardest orientations are those near the [100] pole, consistent with the difficulty of slip in ⟨100⟩ directions for BCC materials as the surface normal orientation approaches the [100] pole. Indentation of Ti-51.5 at percent Ni is elasto-plastic, with a negligible orientation dependence.

2 The macroscopic compressive response of solutionized Ti-50.9 at percent Ni deformed 18 deg above A_f is dependent on the crystallographic orientation of the testing axis. As the normal axis moves towards near the [111] pole, plastic flow dominates. Orientations closer to [210] (near the theoretical maximum Schmid factor for Type II-1 twins) deform mainly by a stress-induced martensitic transformation.

3 The orientation of the ⟨100⟩{001} and ⟨100⟩{011} families of slip systems, with respect to the applied stress-state, strongly influences the development of plasticity in NiTi shape memory alloys. Specimens with crystal orientations that do not favor slip on these systems show better overall recoverability relative to other orientations that are more favorably oriented for the transformation and plastic flow.

4 The strain-hardening of highly symmetric orientations such as [111] or [100] is more pronounced if plastic flow rather than a stress-induced transformation govern the deformation response. This observation indicates that either stress-induced martensite plates interact with low energy, or multiple plates do not form. Slip systems show relatively strong interactions.

Conclusions

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[10] [illegible]

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1 Introduction

Traditional applications of NiTi shape memory alloys include clamps, pipe couplings, actuators, orthodontic wires, blood clot filters, stents, and orthopedic devices [1]. The aforementioned applications exploit the ability of NiTi shape memory alloys to recover inelastic strains on the order of 6 percent by heating (shape memory) or stress removal (pseudoelasticity). We currently understand that a thermo-elastic martensitic transformation from a B2 phase to a monoclinic phase controls the unique thermo-mechanical properties of near equiatomic NiTi when it is deformed near its transformation temperatures [2,3]. Moreover, based on a recent review [4], it is clear that we have a rather solid comprehension of the fundamental macroscopic and microscopic deformation modes resulting from the transformation in NiTi shape memory alloys deformed under monotonic loading. Aside from our breadth of knowledge for NiTi deformed under monotonic loading, we do not have a strong grasp of microscopic deformation mechanisms under cyclic loading. It is surprising that such a deficiency in knowledge exists, despite the fact that NiTi shape memory alloys deform quite differently on the macroscale under cyclic loading conditions [5–23].

Previous experimental work has provided data on the *macroscopic* response of polycrystalline NiTi to pure mechanical [6–11,15] pure thermal [12–15], or thermo-mechanical [15–23] cyclic loading. The aforementioned collection of work provides information on the effects of cycle number, loading path, stress state, stress level, strain level, temperature, and material heat treatment. A common conclusion in all cyclic studies is that the material response generally changes during cycling and the evolution is somehow linked to the interaction between transformation strains and strains due to plastic flow. Only very recent studies have attempted to explicitly uncover the *microscopic* mechanisms for cyclic evolution and degradation effects in NiTi using single crystals [24] and transmission electron microscopy (TEM) [25]. Understanding the microscopic mechanisms of cyclic degradation in NiTi materials is particularly important for emerging shape memory alloys technologies.

Ongoing workshops [26] and conferences [27] have suggested that a lucrative future for shape memory materials such as NiTi lies in the areas of micro-electro-mechanical systems (MEMS) and civil infrastructures. Long-life reliability issues in these applications demand tools to understand and accurately predict the evolution of the NiTi material response under cyclic loading. To improve our state-of-the-art approaches [20–23] aimed at modeling the cyclic behavior of NiTi shape memory alloys, we must increase our mechanistic understandings at the microscopic scale. Moreover, the loading conditions [28–30] and material microstructure [31] in MEMS actuators (usually thin films) are quite different than bulk NiTi components. Consequently, without accounting for the local deformation mechanisms it becomes difficult to extend our macroscopic cyclic models [20–23] to smaller scale material systems without possible deficiencies. In addition, micro-scale studies on NiTi materials are critical to provide a tool for characterization of emerging smart MEMS technologies.

One of the primary reasons that the micro-mechanisms of cyclic evolution and degradation effects have eluded research efforts is because fundamental research on the NiTi alloy system has not explicitly focused on the mechanisms and implications of plastic flow. Ignoring the effects of plastic flow is a viable assumption under monotonic loading since its influence is negligible over one loading cycle. However, under cyclic loading, the local plastic flow characteristics of the B2 matrix have a strong influence on the evolving NiTi material response [24]. During the transformation, the material microstructure is a mixture of martensite, the B2 parent phase, and untransformable precipitates (present in Ni or Ti rich materials). Repeated growth of the martensite through a heterogeneous microstructure causes local slip to accumulate to significant levels in the weaker B2 phase. To our knowledge, there is only one study that examines microscopic slip characteristics in NiTi materials [32], where TEM is used to identify the {100}⟨001⟩ and {100}⟨011⟩ families as dominant slip systems in B2 NiTi. Notably, experimental [33] and atomistic [34] studies in similar B2 NiAl intermetallics have provided consistent findings with the above slip systems for B2 NiTi.

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