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Characterisation of Ni–Ti thin films produced by filtered arc deposition

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Abstract

Ti–49.5 at%Ni thin films have been formed by deposition onto Si and glass substrates using a filtered arc deposition system (FADS). The films deposited on glass were composed of nanocrystalline parent phase grains contained within an amorphous matrix. The films deposited onto silicon were crystalline, and were largely parent phase whereas a bulk alloy of the same composition would be expected to be martensite. The stabilisation of the parent phase is proposed to be a grain size effect, with the critical grain size for parent phase stabilisation being about 30 nm.

Keywords: Shape memory; Thin film; Filtered arc deposition

1. Introduction

The shape memory and pseudo-elastic properties of martensitic Ni–Ti alloys are well documented [1,2]. Shape memory in this system occurs by the competitive growth of thermoelastic martensite variants under stress. The stress-induced martensite, and its accompanying shape change, can be reversed by the reversion of the martensite to the parent phase by stress removal (pseudo-elasticity) or by heating (shape memory) [1]. The parent phase is an ordered bcc crystal structure (B2), and two martensitic transformations can occur, depending on composition and processing history: a trigonal phase (known as R-phase), and a monoclinic martensite known as B19′ [2]. Recently, there has been renewed interest in thin films of these alloys because they have successfully been used as actuators in micro-electro-mechanical devices (MEMS) [3].

Ishida et al. [4] reported the first successful production and characterisation of the shape memory effect in Ni–Ti thin films. The as-deposited films were amorphous, and a crystallization heat treatment was necessary to produce reversible phase transformation and shape memory effect. This work and subsequent investigations have demonstrated that shape memory behaviour equivalent to that of bulk alloys can be obtained in thin films [2,4,5]. Substrate and deposition temperatures have a significant effect on the deposited film. Sputtering produces an amorphous film if the substrate temperature during deposition is below 473 K. Above this temperature, Ni–50 at%Ti films are crystalline parent (B2) phase. In the case of an amorphous film, a post-deposition heat treatment above ∼673 K is sufficient to crystallize the film [1,2].

Most shape memory alloy thin films have been deposited using sputtering [2,3]. However, control of the composition of sputter-deposited films is difficult. In the case of Ni–Ti deposits, Ti depletion occurs relative to the target composition, possibly due to loss by oxidation during deposition [5]. Another problem encountered during sputtering is that the composition of the deposited films is not always uniform across the entire width of the substrate [5]. As well as the presence of significant micro-defects in the form of fine pores, sputter-deposited films are subject to contamination by interstitial oxygen and hydrogen atoms, and brittle fracture can result on the application of stress [3]. Nevertheless, RF magnetron sputtering has become the default option for film production because of even greater shortcomings (particularly, homogeneity) of films produced by other physical vapour deposition techniques [6]. The control of composition is critical in this alloy system to prevent detrimental precipitation of, for example, Ni_3Ti_4 [1,2]. This sensitivity to composition requires that the film is homogenous, and that the composition of the film is predictable.

Instead of using magnetron sputtering, the films examined in this work have been deposited using a filtered arc deposition system (FADS). Like sputtering, FADS is a physical

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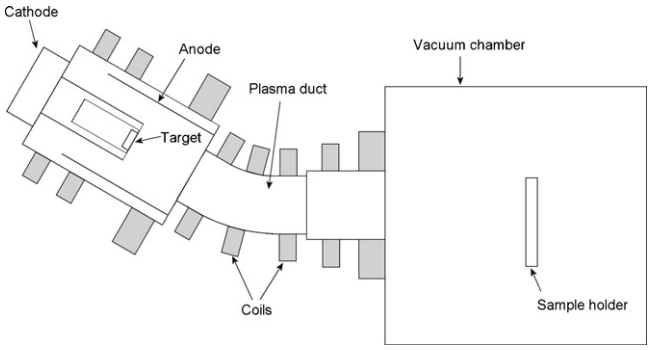

Fig. 1. Schematic of the filtered arc deposition system used to deposit the thin films.

vapour deposition technique. However, FADS is an evaporation, rather than a sputtering, process in which the thermally activated release of target ions/atoms is achieved by a cathodic arc source [7]. This plasma is collimated through a curve or S-bend by a magnetic field to filter out any droplets of solid or liquid that have been ejected from the arc spot [8,9]. This filtered vapour plasma is then deposited onto the substrate.

Direct comparison between magnetron sputtering and FADS for the deposition of metal layers of Ti and TiN has shown that FADS produces a less porous film with superior chemical homogeneity [10]. It is also demonstrated in the current report that films of the same composition as the target material can be produced using FADS. This overcomes the main disadvantage of sputtering.

In this paper we report on the composition, microstructure and thermal transformation behaviour of Ni–Ti films deposited by FADS. The mechanical properties and shape memory behaviour of the films will be the subject of another publication.

2. Experimental method

Thin films of binary Ni–Ti were deposited using a filtered arc deposition system (FADS). A schematic diagram of the FADS used is shown in Fig. 1. The FADS employs a vacuum arc as a source of energetic, ionized material, and macroparticles ejected from the arc spot are filtered by a curved plasma duct. Deposition is carried out at a relatively high vacuum, 5×10^{-5} Torr, and is typically operated at a beam current of 0.7–0.8 A and a bias of between 20 and 100 V for 10–20 min. A more detailed description of the FADS is given elsewhere [11]. The target material for the FADS was purchased from a commercial supplier and was received in the as-cast condition. The composition of the target is given in Table 1.

Table 1
Composition of target material

Ni	49.8 at%
Ti	Balance
C	280 ppm
O	189 ppm

Two groups of films were produced. The first group were deposited onto (1 1 1) Si wafers under various deposition conditions to examine the effect of deposition current on the composition of the film. Once the deposition parameters were optimized, the second group of films were prepared. The second group of films comprised three different substrates: glass, (0 0 1) Si single crystal wafer, and a (1 1 1) single crystal Si wafer. Microstructural examination was only carried out on the second group. Other than the substrate type, the deposition conditions were identical for each of the three samples in the second group. The substrate temperature during deposition was approximately 373 K. This temperature results only from the deposition process, and no additional heating was supplied to the substrate. Films in the second group were deposited for 20 min, had a thickness of 2.4 μm, and a composition of Ti–49.5 at%Ni. This composition measurement was made using an Oxford Instruments energy dispersive spectroscopy system fitted to a Leica 440 scanning electron microscope. X-ray diffraction (XRD) was carried out at a scan rate of 2° min^{-1} using Cu Kα radiation. To examine the transformation temperatures of the films, differential scanning calorimetry (DSC) was conducted by using a TA Q100 differential scanning calorimeter. The sample size for DSC analysis was between 1.5 and 4 mg, and samples were hermetically sealed into aluminium pans before DSC scanning. A JEOL FX 2011 transmission electron microscope (TEM) was used to examine the microstructure of the films at an accelerating voltage of 200 kV. Samples were prepared for TEM using a Gatan plasma ion polishing system.

3. Results

3.1. Optimization of deposition parameters

The first group of samples was prepared by depositing films onto (1 1 1) silicon wafers. These films were deposited with a deposition bias of 100 V. The effect of deposition current on the films composition is shown in Fig. 2. Increasing the deposi-

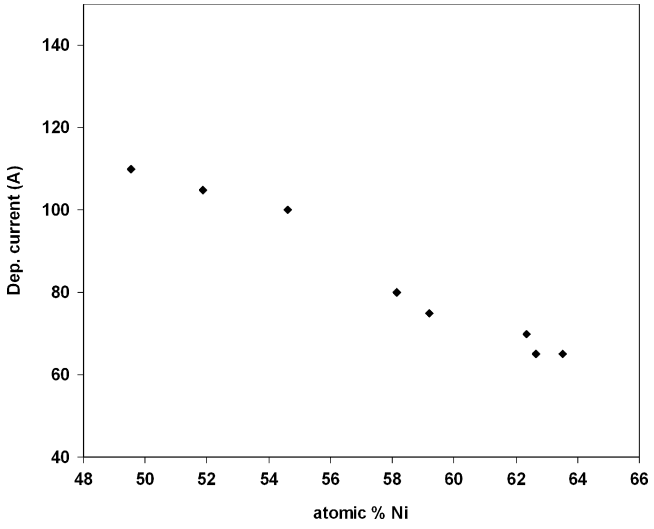

Fig. 2. Effect of deposition current on the composition of films deposited using FADS.