The effect of interfaces number on solid state reactions in Ni-Ti thin films

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Introduction

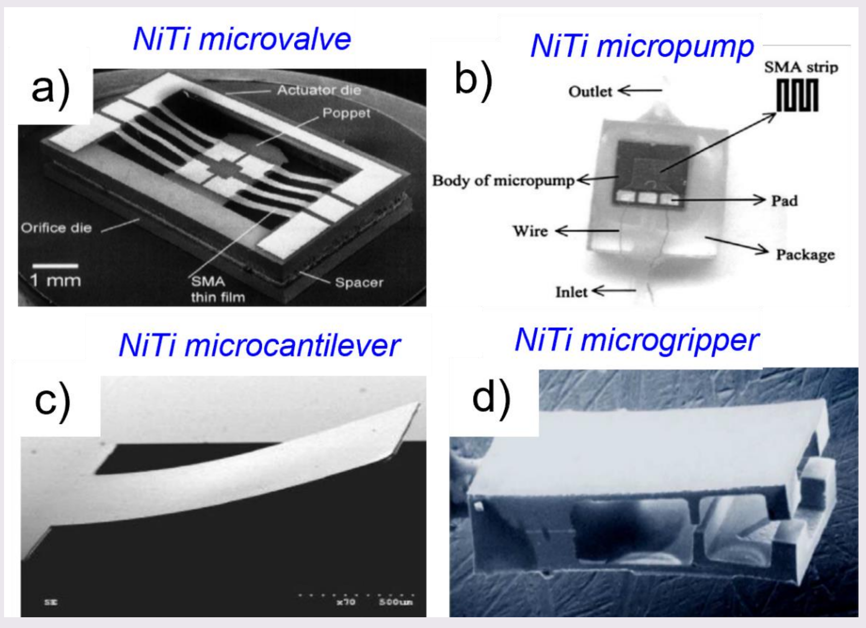

Fig. 1. MEMS microactuators based on NiTi [1]

NiTi shape memory thin films are highly attractive materials for microelectromechanical systems (MEMS). The actuality of thin-film NiTi is attributed to its large recovery strain and high work output per unit, being useful for the fabrication of microactuators, microsensors, micropumps etc. (Fig. 1). The common pathway for NiTi fabrication is the film sputtering from the NiTi alloy target. However, the alternative approach is the deposition of layered stack with Ni and Ti individual layers, which is helpful in terms of more precise tuning of the chemical composition of the equiatomic intermetallic phase (Fig. 2).

Upon the post-deposition annealing, the intermetallic compound is typically formed through the stages of amorphization and next crystallization [2]. However, in this case the number of the layers involved in the initial stack could drastically influe the solid-state reactions kinetics due to the crucial role of interfaces on the diffusion in thin films.

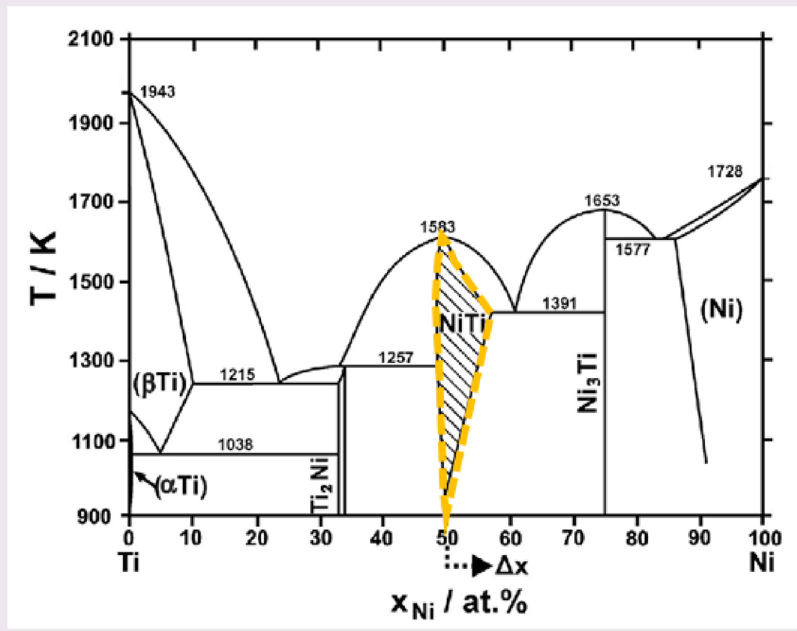

Fig. 2. Ni-Ti binary phase diagram

Objects and Methods

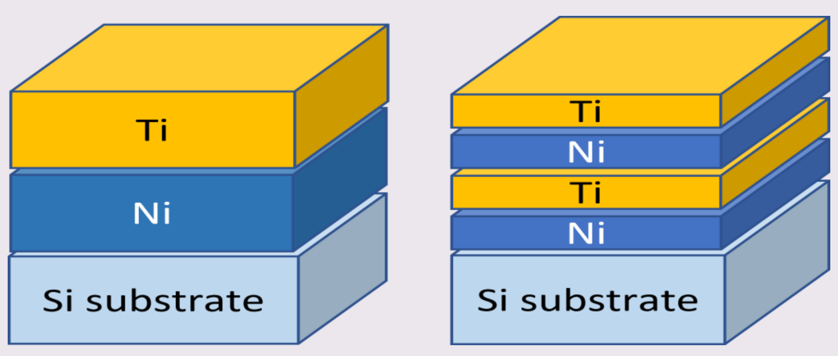

Fig. 3. Ni/Ti bi-layered and four-layered structures

Present study is aimed to figure out the effect of interfaces number on the solid-state reactions taking place upon annealing in high vacuum (10^{-4} Pa) for 30 min of Ni(30 nm)/Ti(30 nm) and $[Ni(15\ nm)/Ti(15\ nm)]_{2x}$ thin films (Fig. 3) prepared onto Si(001) substrate using rf magnetron sputtering at room temperature. Post-deposition thermal annealing of thin-film samples was performed in vacuum (10^{-4} Pa) in the temperature range 300-700^{o}C for 30 minutes.

Experimental results

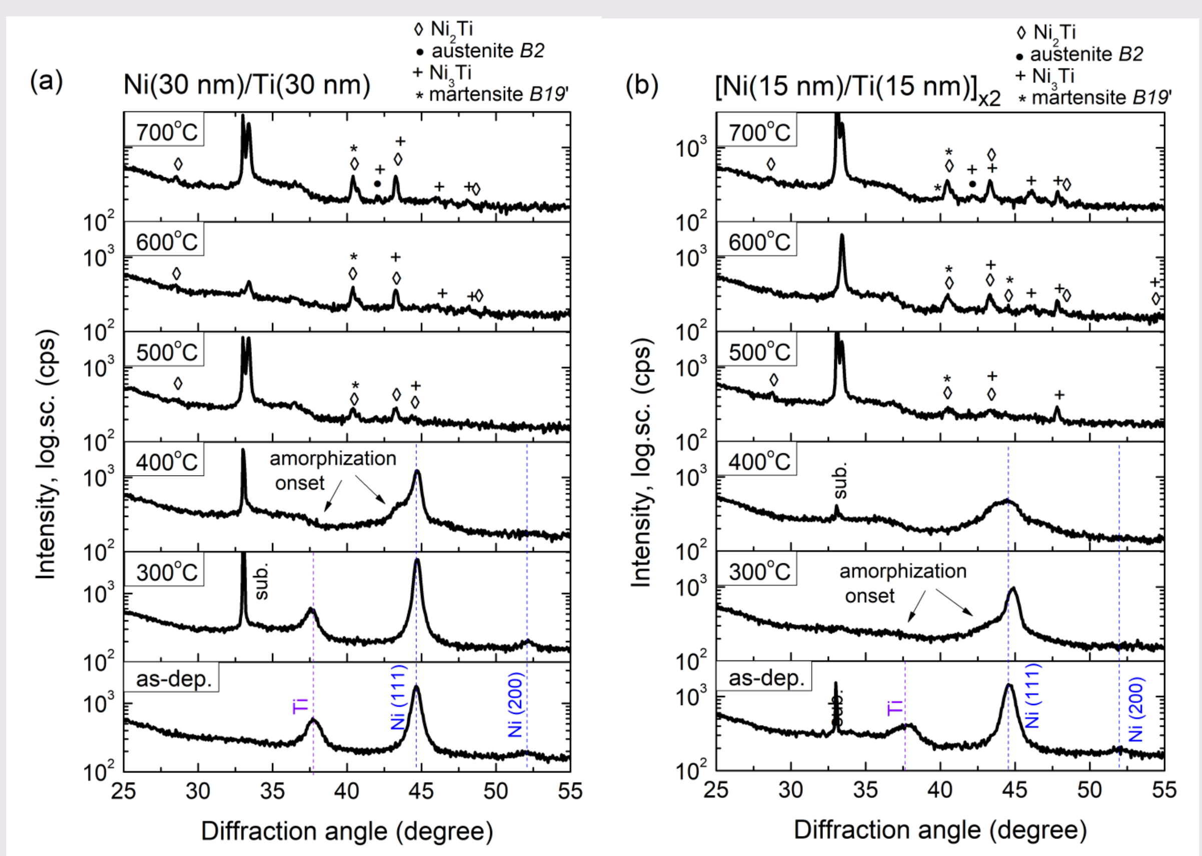

Fig. 4. XRD scans of Ni/Ti bi-layered (a) and four-layered (b) samples after annealing

Structural study performed using XRD technique revealed (Fig. 4) the difference in the amorphization behavior at the first stages of annealing of two- and four-layered stacks. It was found that the onset temperature of both amorphization and crystallization is about 100^{o}C lower in the film consisted of four layers compared to the bilayer system. The following rise of the temperature resulted in the similar recrystallization behavior with the formation of the NiTi intermetallic compounds observed for both films.

This results was further supported by the data from GIWAXS scanning (Fig. 5). Amorphization onset as well as all the solid-state reactions take place at lower temperature when the larger number of internal interfaces and smaller layer thickness are used. Furthermore, the GIWAXS data indicated formation of TiO_2 oxide with a strong texture after annealing at 500^{o}C, which indicates on the diffusion of Ti on the outer surface followed by its oxidation.

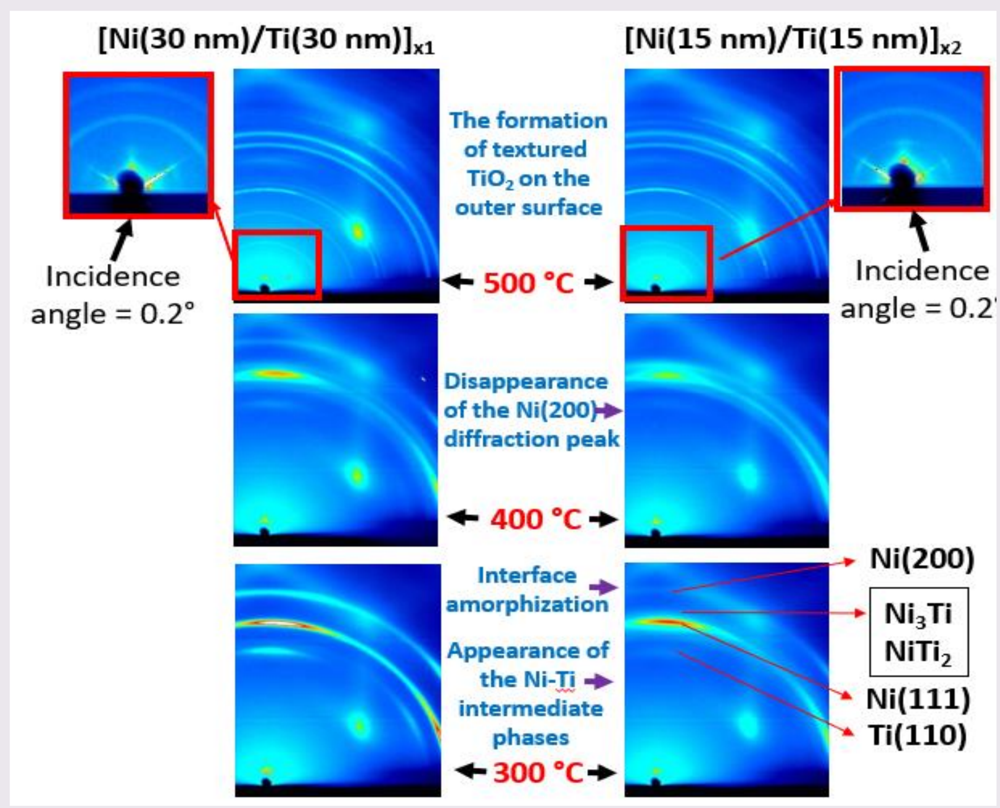

Fig. 5. GIWAXS scans of Ni/Ti bi-layered (a) and four-layered (b) samples after annealing

To verify the surface composition modification from Ni to Ti, additional XPS measurements were carried out. XPS data clearly has confirmed (Fig. 6), that upon annealing, Ti diffuses to the outer surface and forms Ti(IV) TiO_2 oxide layer. As a result, the peaks from Ni disappear. Furthermore, the larger number of internal interfaces leads to the formation of Ti(III) Ti_2O_3 oxide in addition to TiO_2. The results of AFM measurement (Fig.7) of surface morphology as a function of temperature has revealed that the RMS roughness of bi- and four-layered Ni/Ti films has a similar temperature behavior. While for the as-deposited films the roughness is below 0.5 nm, it starts drastically increasing (to 6 nm) after high-temperature annealing (>500^{o}C).

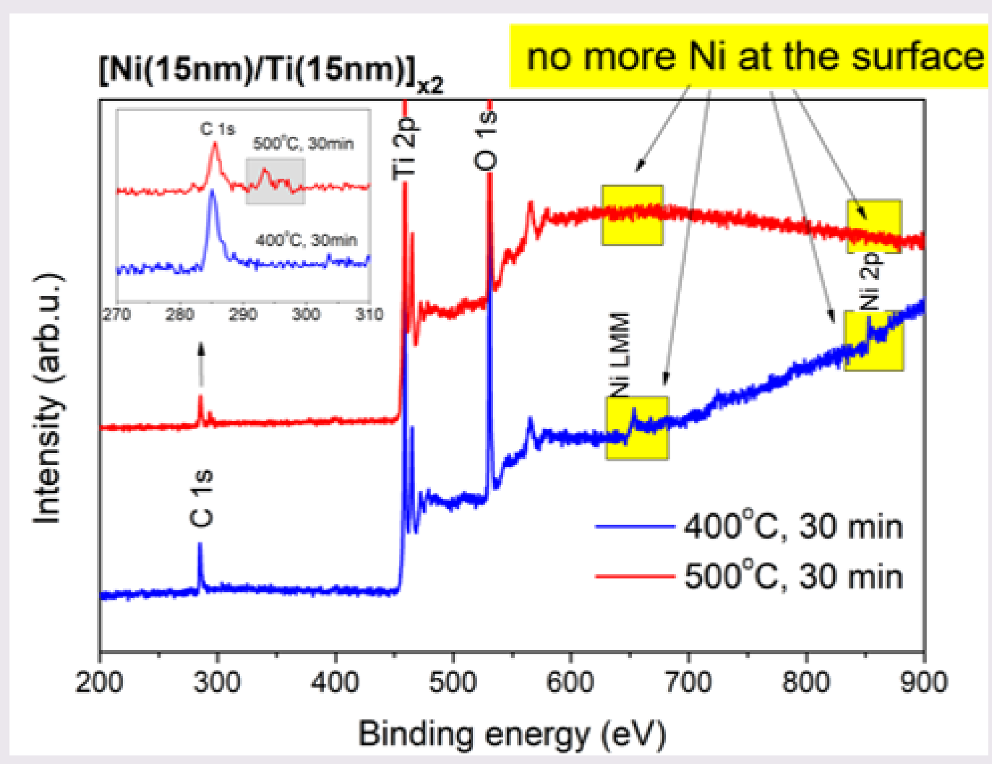

Fig. 6. XPS scan of Ni/Ti thin films after thermal annealing

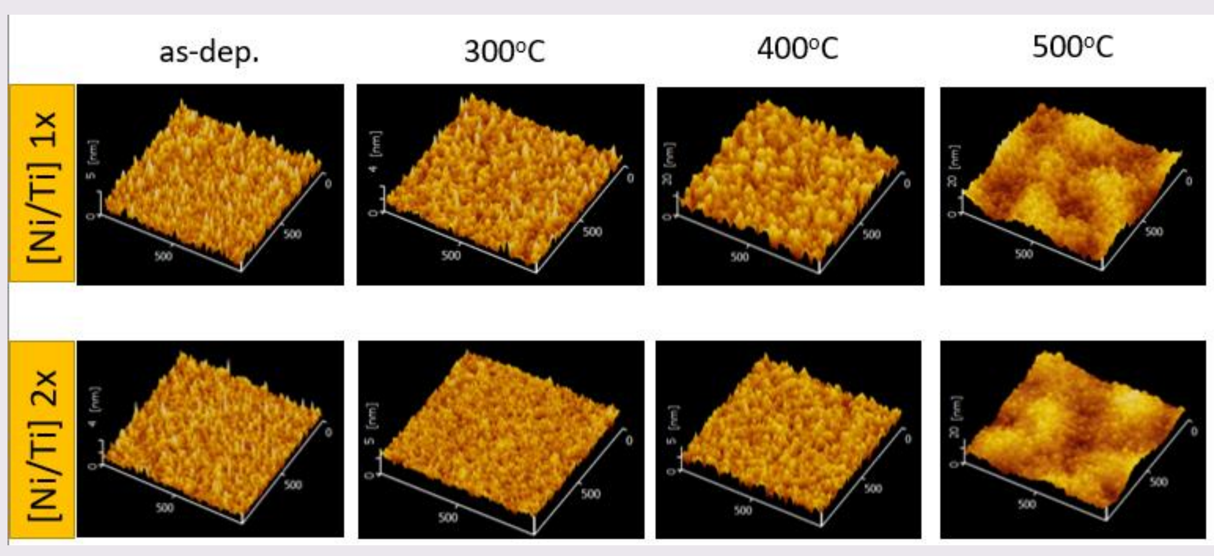

Fig. 7. Surface morphology of Ni/Ti bi-layered and four-layered samples after annealing

Conclusions

In present study the thermally-induced solid state reactions were studied in Ni/Ti thin-film stacks with a different number of individual layers (two vs four) but the same total stack thickness of 60 nm. The results of structural data obtained from XRD and GIWAXS indicated that the amorphization of the interfaces is taking place at 300^{o}C. The annealing temperature increase up to 500^{o}C lead to formation of the TiO_2 protective oxide layer on the outer surface. All solid state reactions are accelerating and occur at 100^{o}C lower temperatures in the case of four-layered stack $[Ni/Ti]_{x2}$.

References

[1] Choudhary N. and Davinder K. Shape memory alloy thin films and heterostructures for MEMS applications: A review, Sensors and Actuators A: Physical 242 (2016): 162-181.

[2] Lehnert T. et al. Characterization of shape-memory alloy thin films made up from sputter-deposited Ni/Ti multilayers, Acta mater. 48 (2000) 4065.

Acknowledgments

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