STRUCTURAL AND MORPHOLOGICAL NATURE OF Ti-Si LAYERS

Petr VLCAK, Vidzaja KNAP, Vojtech SMOLA, Jan DRAHOKOUPIL, Jan BARTONICEK

Czech Technical University in Prague, Faculty of Mechanical Engineering, Czech Republic, EU

https://doi.org/10.37904/nanocon.2025.5191

Abstract

The structure and morphology of binary Ti-Si thin films were studied. Ti-Si surface alloys were deposited by electron beam. The Si content was controlled by the evaporation rate of Ti and Si targets. The structure was investigated by X-ray diffraction (XRD) and the morphology and surface roughness were measured by atomic force microscopy (AFM). The results show that the morphology and surface roughness reflect the structural evolution. The crystalline nature of Ti-Si thin films tends to nanocrystalline form. Higher amounts of Si resulted in an amorphous structure with a smoothed surface morphology and the lowest roughness.

Keywords: Ti-Si surface alloys, structure, surface morphology, roughness

1. INTRODUCTION

In recent years, Thin Film Metallic Glasses (TFMGs) have garnered significant attention in materials science due to their unique amorphous structure and resulting properties [1]. Unlike traditional crystalline materials, TFMGs lack grain boundaries, which imparts them with high strength, excellent corrosion resistance, and remarkably smooth surfaces [2]. These characteristics make them highly desirable for a range of applications, including biomedical devices and sensors [3]. The pursuit of new compositions and fabrication methods to achieve and control the amorphous state in thin films is a prominent area of ongoing research [4, 5]. Among the various material systems (monolayer and multilayer systems [6, 7]) being explored, simple binary alloys present an attractive route for producing TFMGs [8, 9]. The titanium-silicon (Ti-Si) system, in particular, is a compelling choice for developing these advanced coatings [8]. Titanium and its alloys are renowned for their superior biocompatibility and non-toxic nature, making them a cornerstone for medical implants [10, 11]. By introducing silicon, it is possible to disrupt the crystalline structure of titanium and induce a transition to a nanocrystalline and, with sufficient Si content, a fully amorphous state [8, 12]. This ability to tailor the structure from crystalline to amorphous in a controlled manner within a straightforward binary system is a key advantage. This study focuses on the structural and morphological evolution of Ti-Si thin films as a function of composition.

2. EXPERIMENTAL PART

Silicon wafers (10×10×1 mm) with crystallographic orientation 111 were used as substrates. Three Ti-Si layer types with various Si content were prepared (samples 95Ti-5Si, 85Ti-15Si and 80Ti-20Si). The deposition was performed by simultaneous electron beam evaporation of Ti and Si targets. The structure of Ti-Si surface alloys was analyzed using X-ray diffraction (XRD). XRD was used to investigate the structural evolution depending on the content of Si. The XRD measurement was conducted using an X'Pert PRO diffractometer with a Co anode, parallel beam geometry, a parallel beam mirror in the incident beam, and a parallel beam collimator in the difracted beam (with an acceptance of 0,09°). The incident angle was set at 3°. The surface topography was characterized using an AFM NanoWizard3 instrument (JPK, Berlin, Germany). Measurements were performed in contact mode in air, with a scanning rate of 1 Hz and setpoint of 2.30 V. The scanned areas were 1×1 μm^2, with resolution of 256×256 pixels. To correct for sample tilt, all topography images were flattened using the plane fitting option in the JPK data processing software. The surface roughness was determined as the average of the RMS roughness (Rq) and the arithmetic roughness (Ra) values obtained from three independent areas.

3. RESULTS

The structural evolution of the Ti-Si films with varying compositions was investigated using X-ray Diffraction (XRD), and the patterns are shown in **Figure 1**. The diffractogram for the $Ti_{95}Si_5$ sample, displays several sharp and well-defined diffraction peaks, which is characteristic of a polycrystalline material. For the $Ti_{85}Si_{15}$ sample, the peaks become significantly broader and their intensity is reduced. This peak broadening is a clear indication of a reduction in grain size, signifying a transition to a nanocrystalline structure. Finally, the top diffractogram, corresponding to the $Ti_{80}Si_{20}$ sample with the highest silicon content, shows a complete absence of sharp crystalline peaks. Instead, a single broad halo or "hump" is observed, which is the distinctive signature of an amorphous, or glassy, structure. These results demonstrate a clear structural progression from a crystalline to a nanocrystalline and ultimately to an amorphous state as the silicon content in the Ti-Si thin films is increased.

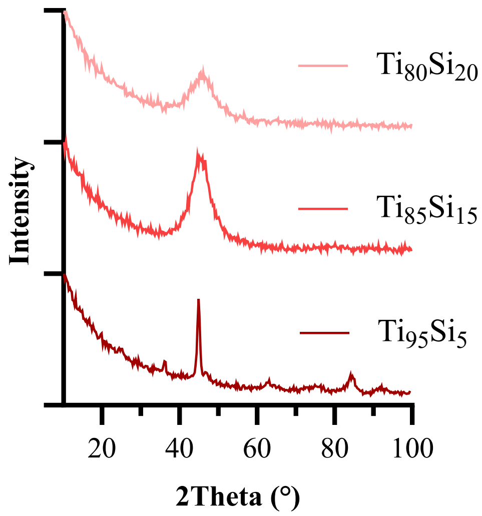

Figure 1 Diffractograms of $Ti_{80}Si_{20}$, $Ti_{85}Si_{15}$ and $Ti_{95}Si_5$ samples

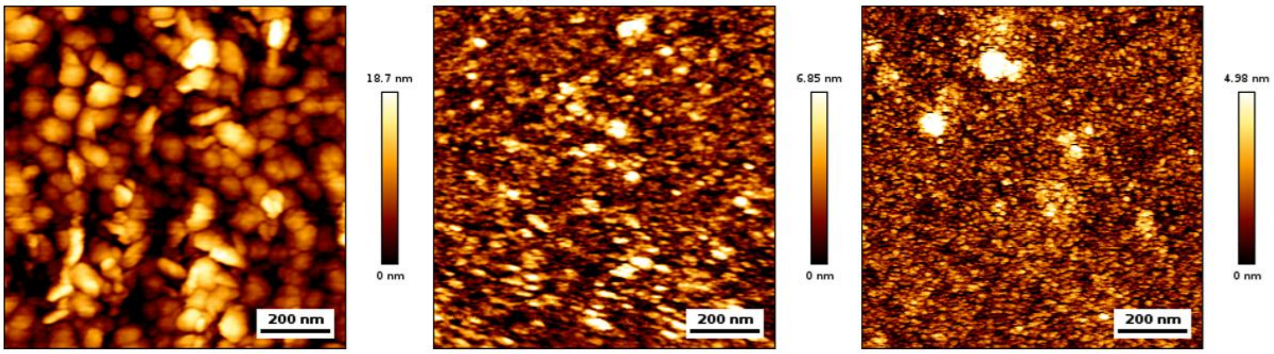

Figure 2 AFM images of of Ti-Si thin films: $Ti_{95}Si_5$ (left), $Ti_{85}Si_{15}$ (middle) and $Ti_{80}Si_{20}$ (right). The height topography images were recorded for an area 1×1 μm^2

The surface morphology and topography of the Ti-Si thin films were investigated using Atomic Force Microscopy (AFM), with the results presented in **Figure 2**. The images and the quantitative roughness analysis reveal a strong correlation between the surface morphology and the underlying structure identified by XRD.

The AFM image of the crystalline $Ti_{95}Si_5$ film displays a coarse surface composed of large, distinct, and irregularly shaped grains. This structure results in the highest surface roughness, with an average roughness (Ra) of 3.4 nm and a root-mean-square roughness (Rq) of 4.3 nm, as detailed in **Table 1**. In contrast, the morphology of the nanocrystalline $Ti_{85}Si_{15}$ film is markedly different. The surface features become much smaller and more uniform, leading to a significant reduction in roughness, with Ra and Rq values decreasing to 0.8 nm and 1.0 nm, respectively. This smoothing trend culminates in the amorphous $Ti_{80}Si_{20}$ film. As confirmed by its AFM image, the surface is nearly featureless, lacking any discernible grain structure. This homogeneous and smooth topography, a direct consequence of its amorphous nature, corresponds to the lowest measured roughness values of Ra = 0.7 nm and Rq = 0.9 nm. This progression, clearly visualized in **Figure 3**, demonstrates that increasing the silicon content not only induces a structural transition from crystalline to amorphous but also systematically refines the surface morphology, leading to a substantially smoother film.

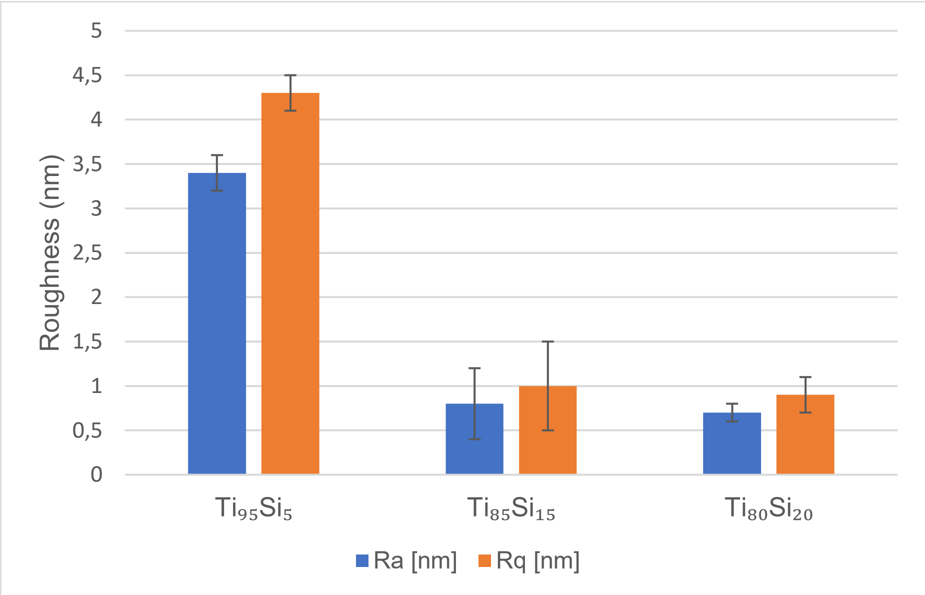

Figure 3 Average surface roughness values with standard deviations for the prepared Ti-Si films

Table 1 Surface roughness parameters (Ra and Rq) for the Ti-Si films from 1×1 μm² areas.

	$Ti_{95}Si_5$		**$Ti_{85}Si_{15}$**		**$Ti_{80}Si_{10}$**	
Number	Ra [nm]	Rq [nm]	Ra [nm]	Rq [nm]	Ra [nm]	Rq [nm]
1	3.2	4.1	1.2	1.6	0.8	1.1
2	3.5	4.4	0.5	0.7	0.7	0.9
3	3.6	4.4	0.6	0.7	0.6	0.8
Average	**3.4**	**4.3**	**0.8**	**1.0**	**0.7**	**0.9**
STDEV	0.2	0.2	0.4	0.5	0.1	0.2

4. CONCLUSION

The structural and morphological analysis of Ti-Si thin films revealed a clear dependence on silicon content. XRD measurements showed a systematic structural evolution from crystalline ($Ti_{95}Si_5$), through nanocrystalline ($Ti_{85}Si_{15}$), to fully amorphous structure ($Ti_{80}Si_{20}$). AFM characterization confirmed that this transition is accompanied by a progressive refinement of surface morphology. Higher silicon content led to significantly smoother films, with roughness decreasing from Ra = 3.4 nm in the crystalline film to Ra = 0.7 nm in the amorphous film. These results demonstrate that the amorphous state can be achieved in a simple binary Ti-Si system via electron-beam co-evaporation, and that the resulting amorphous films exhibit superior surface smoothness.

ACKNOWLEDGEMENTS

This research has been supported by the Ministry of Education, Youth and Sports of the Czech Republic (Czech Technical University in Prague - SGS24/121/OHK2/3T/12).

REFERENCES

[1] YIU, P., DIYATMIKA, W., BÖNNINGHOFF, N., LU, Y. C., LAI, B. Z., & CHU, J. P. *Thin film metallic glasses: Properties, applications and future.* Journal of Applied Physics. 2020, vol. 127, no. 3, 030901.

[2] DIYATMIKA, W., CHU, J. P., KACHA, B. T., YU, C. C., & LEE, C. M. *Thin film metallic glasses in* optoelectronic, magnetic, and electronic applications: A recent update. Current Opinion in Solid State and Materials Science, 2015, vol. 19, no. 2, pp. 95-106.

[3] RAJAN, S. T., & AROCKIARAJAN, A. (2021). *Thin film metallic glasses for bioimplants and surgical tools: A review.* Journal of Alloys and Compounds, 2020, vol. 876, 159939.

[4] MA, X., SUN, K., LI, P., ZHANG, N., WANG, Q., & WANG, G. *Enhanced mechanical properties of metallic glass thin films via modification of structural heterogeneity.* Materials, 2021, vol. 14, no. 4, 999.

[5] VLCAK, P., HORAZDOVSKY, T., VALENTA, R., KOVAC, J. *Evolution of the nitrogen depth distribution in an implanted titanium alloy with a surface carbon nanolayer.* Chemical Physics Letters, 2017, vol. 679, pp. 25-30.

[6] ANTON, R., HÜNIG, S., LASKA, N., WEBER, M., SCHELLERT, S., GORR, B., CHRIST H.J., HEILMAIER, M., SCHULTS, U. *Interface reactions of magnetron sputtered Si-based dual layer coating systems as oxidation protection for Mo-Si-Ti alloys, Surface & Coatings Technology, 2022, vol. 444, 128620.*

[7] QIAO, L., YANG, Z.B., LI, X.W., HE, D.Y. *Improvement of electrochemical performances of ultrathin* Ti-*coated* Si-*based* multilayer *nanofibers as anode materials for lithium-ion batteries, Surface & Coatings Technology, 2021, vol.424, 127669.*

[8] CAI, Z., DU, P., LI, K., CHEN, L., & XIE, G*. A review of the development of titanium-based and magnesium-based metallic glasses in the field of biomedical materials.* Materials, 2024, vol. 17, no. 18, 4587.

[9] WANG, D., LI, Y., SUN, B. B., SUI, M. L., LU, K., & MA, E. *Bulk metallic glass formation in the binary Cu–Zr system*. Applied Physics Letters, 2004, vol. 84, no. 20, pp. 4029-4031.

[10] ZHANG, L. C., & CHEN, L. Y. *A review on biomedical titanium alloys: recent progress and prospect.* Advanced Engineering Materials, 2019, vol. 21, no. 4, 1801215.

[11] AZMAT, A., ASRAR, S., CHANNA, I. A., ASHFAQ, J., ALI CHANDIO, I., CHANDIO, A. D., DEVANESAN, S. Comparative study of biocompatible titanium alloys containing non-toxic elements for orthopedic implants. *Crystals*, 2023, vol. 13, no. 3, 467.

[12] CHEN, P. S., TSAI, P. H., LI, T. H., JANG, J. S. C., HUANG, J. C. C., LIN, C. H., LIN, H. K. *Development and fabrication of biocompatible Ti-based bulk metallic glass matrix composites for additive manufacturing.* Materials, 2023, vol. 16, no. 17, 5935.