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«RECENT ADVANCES in SCIENCE and TECHNOLOGY»
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The mechanical properties of Ti-Ni-Ta- based surface alloys on the NiTi-substrate formed by the additive thin-film electron beam synthesis

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The structure

1. Introduction

1. What are metallic glasses?
2. Application and problematic fields
3. Surface alloys

2. Materials and methods

1. NiTi shape memory alloy
2. Additive thin-film electron beam synthesis
3. Instrumented indentation

3. Results and discussion

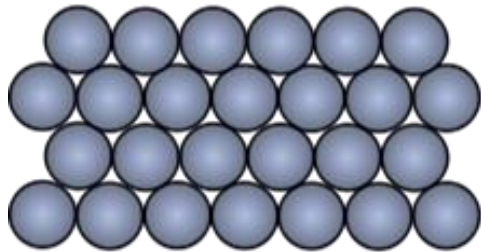
4. Conclusion
5. References
6. Acknowledgments



METALLIC GLASSES

*Comparative characteristics
of crystalline and amorphous state*

CRYSTALLINE STRUCTURE



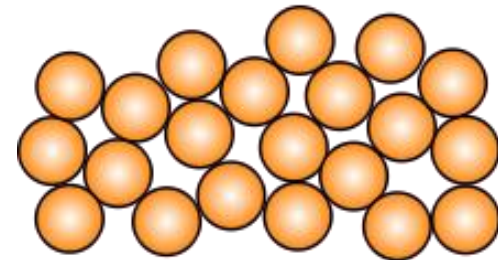
Long atomic order
Translational symmetry

Structural anisotropy

High elastic and plastic properties

Strain hardening

AMORPHOUS STATE



Near atomic order
Disordered structure

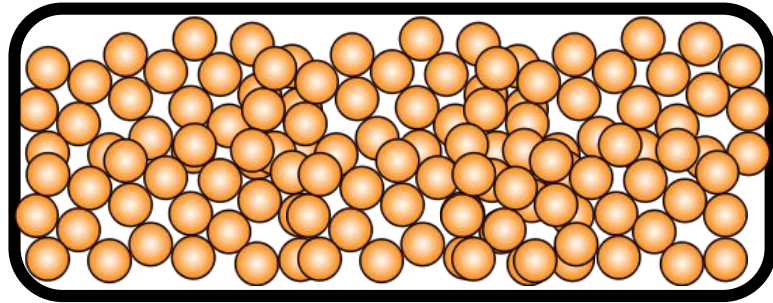
Isotropic material

Low plastic properties

No strain hardening 

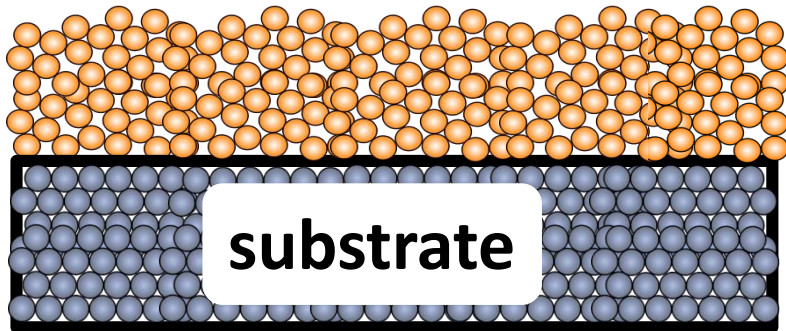
METALLIC GLASSES

Introduction



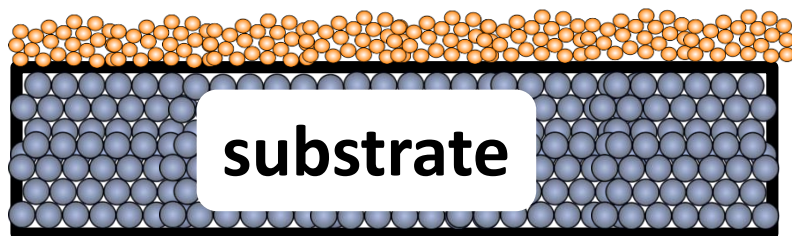
BULK METALLIC GLASSES

(BMGs) ($h \geq 10$ mm)



AMORPHOUS COATING on the metallic substrate

($10 \mu\text{m} \leq h \leq 100 \mu\text{m}$)



THIN-FILM METALLIC GLASSES

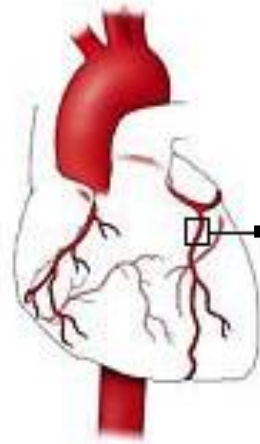
(TFMGs) ($h \leq 100 \mu\text{m}$)



NiTi stent



Stenosis of artery



Deformed shape



T ↑



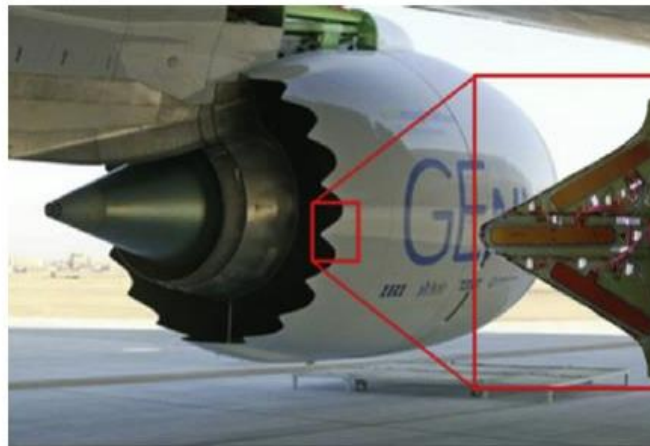
Original shape



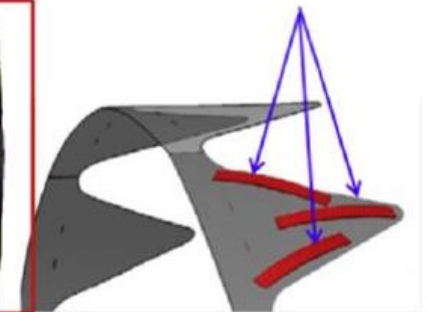
Springs / Wires

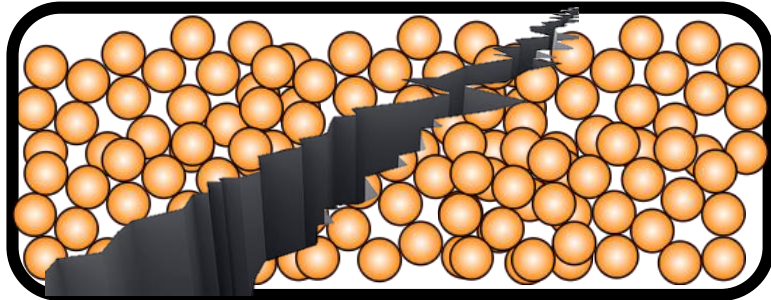


Boeing's variable geometry of nozzle



Shape memory elements

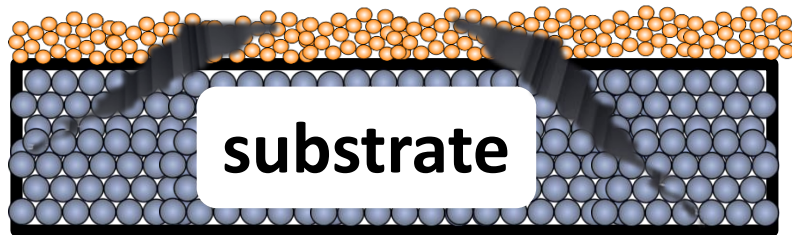
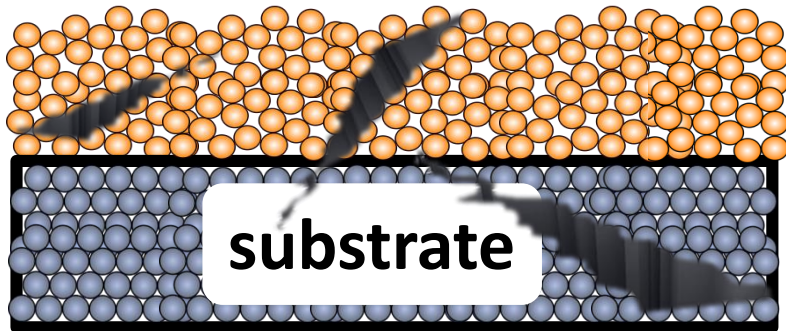




CRACK PROPAGATION

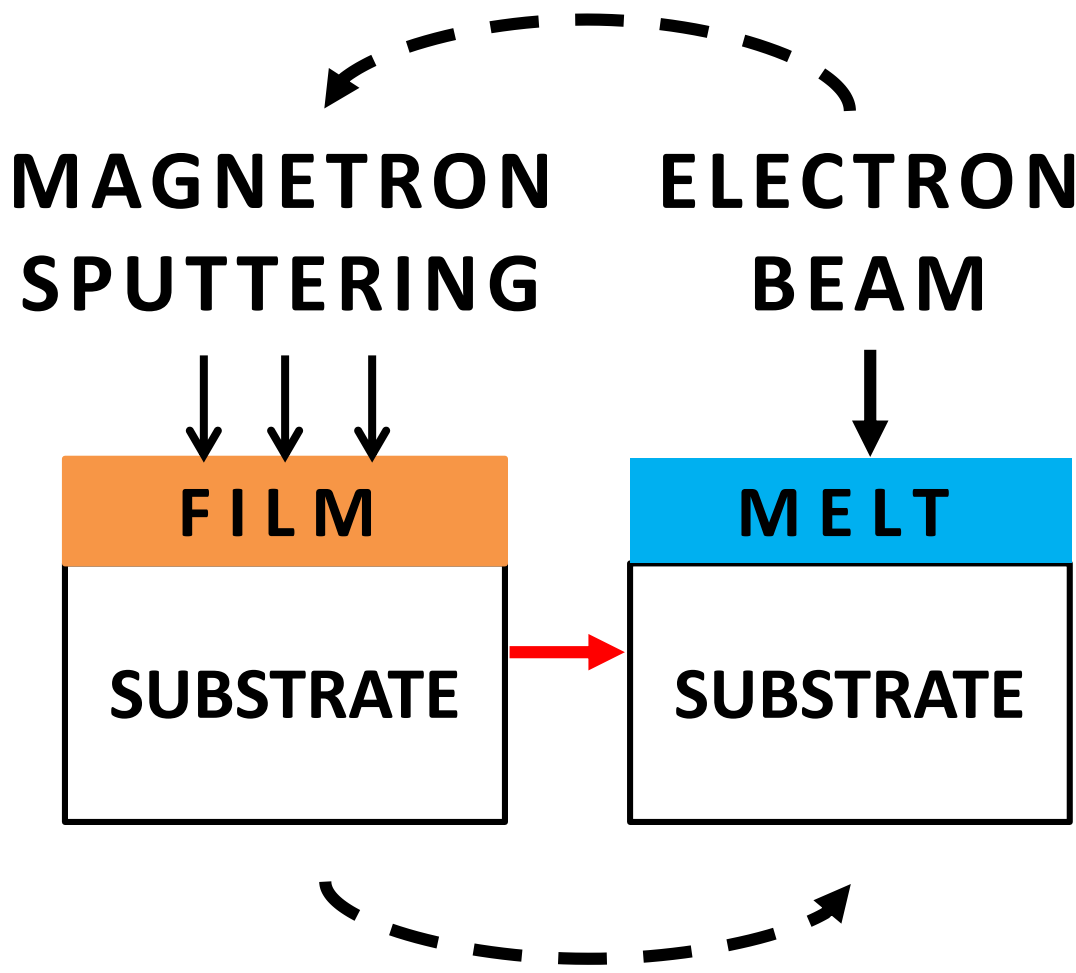
due to the

low ductility of
amorphous state



SURFACE ALLOYS

Additive thin-film electron beam synthesis



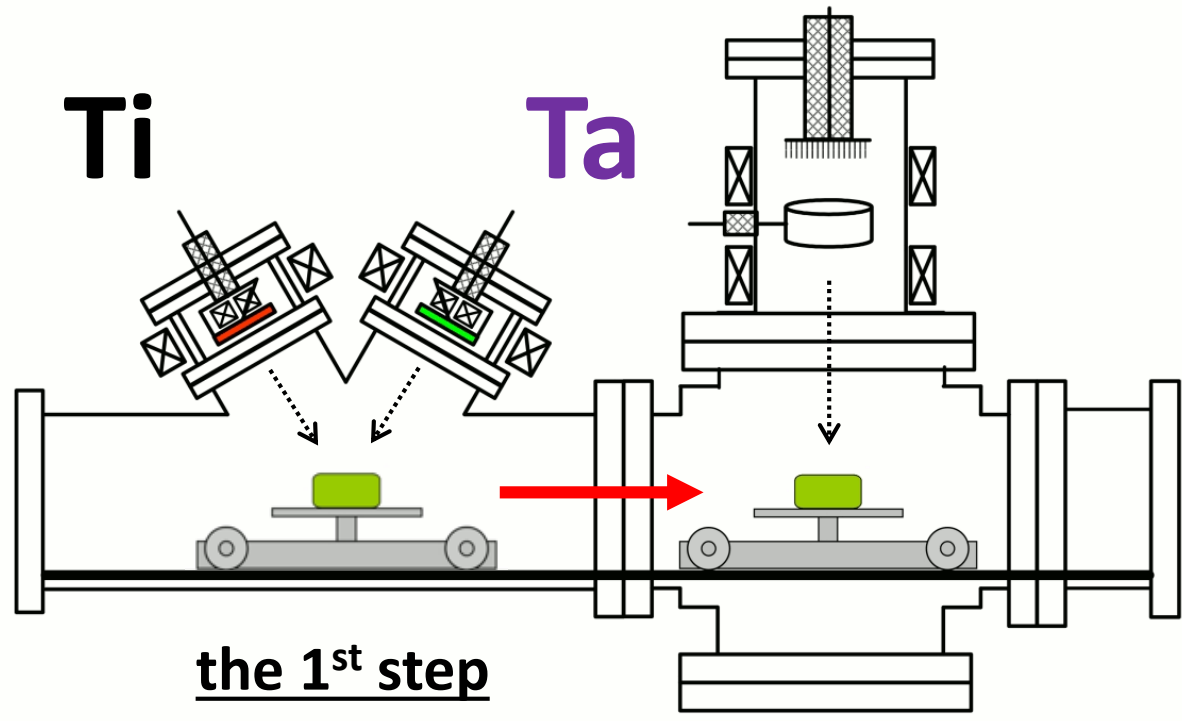
Additive thin-film electron beam synthesis

**MAGNETRON
SPUTTERING**

**ELECTRON
BEAM**

Ti

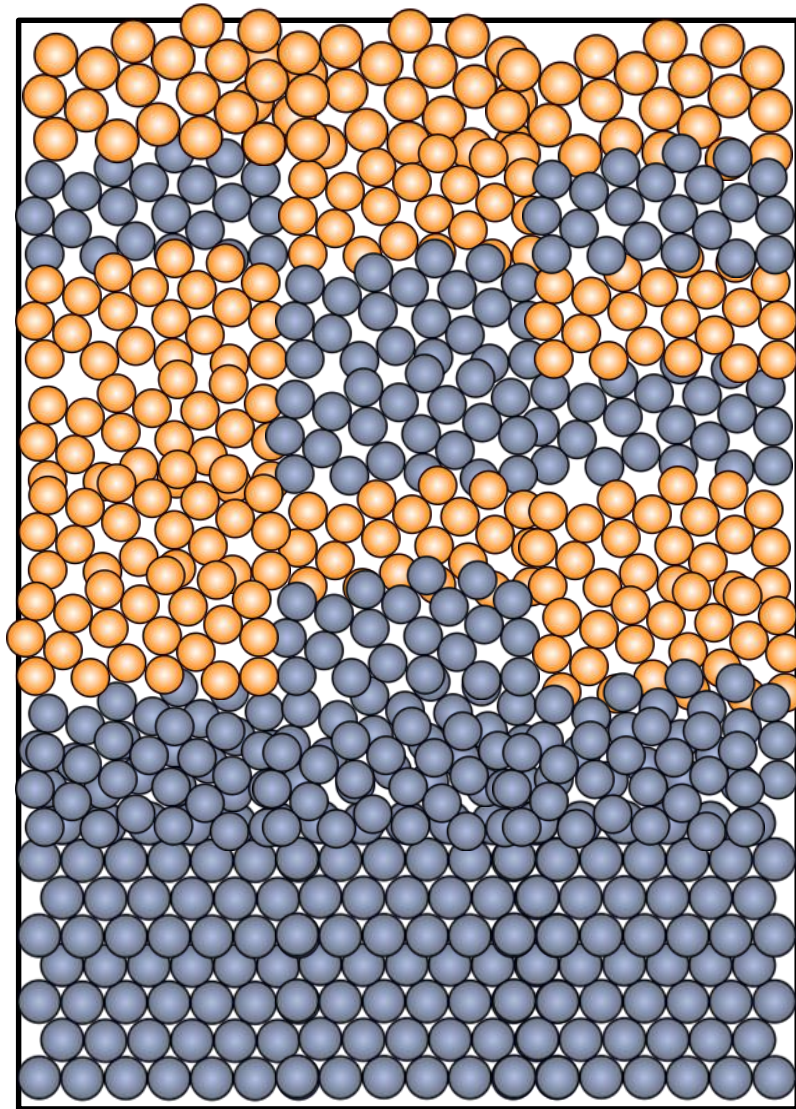
Ta



the 1st step

the 2nd step





**SURFACE
ALLOY**

SUBSTRATE



to investigate the mechanical properties of
Ti-Ni-Ta-based surface alloys on the NiTi-substrate
formed by the additive thin-film
electron beam synthesis

The scientific and applied tasks

- to study basic principles and methods of investigations of mechanical properties of the materials on submicro- and microscale levels
- to investigate the mechanical properties of the surface layers of the NiTi samples before and after additive thin-film electron beam synthesis by method of the instrumented indentation
- to determine the values of hardness H , elastic modulus E , characteristic of plasticity δ and recovery ratio η of the indent on the synthesized layers ★

Materials and methods

Commercial *NiTi alloy* produced as rolled sheets by vacuum induction melting (MATEK-SMA, Russia)

Chemical composition:

Ti – 55.08Ni – 0.051C – 0.030O – 0.002N (wt. %)

Specimen's size – 10 x 10 x 1 mm

Preliminary surface treatment

1
Chemical etching

In acid bath
(3 p. HNO₃ + 1 p. HF)

2
Electrolytically
polishing

In acid bath
(3 p. CH₃COOH + 1 p. HClO₄)
and ice-water mixture

3
Ultrasonic bath

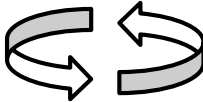
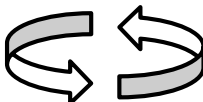
Three times in
distilled water

4
LEHCEB*
treatment

Energy density
 $E_s = 3,4 \text{ J/Cm}^2$
Pulses
 $n = 32$ ★

LEHCEB – low-energy high-current electron beam*

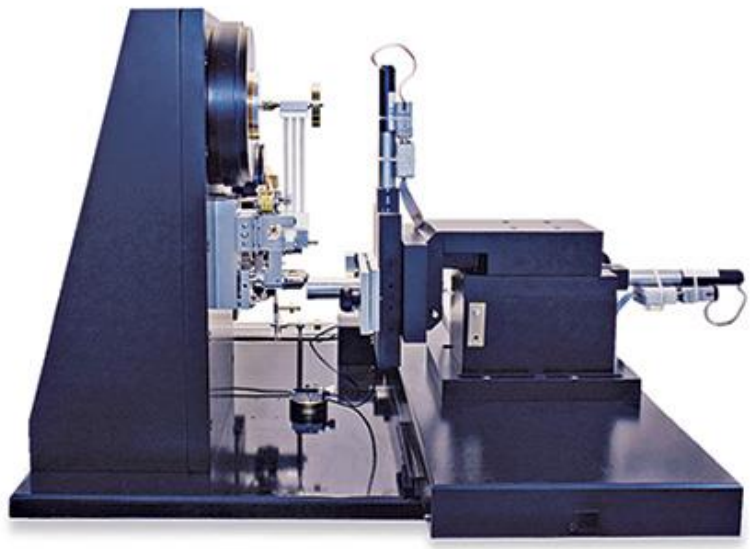
Additive thin-film electron beam synthesis

#	Magnetron sputtering	Pulsed electron beam melting of "film-substrate" system	Thickness $h, \mu\text{m}$
1	$\text{Ti}_{70}\text{Ta}_{30}$ 50 nm	 $E_s = 2 \text{ J/cm}^2$ $n = 5$ Number of cycles $N = 20$	~1
2	$\text{Ti}_{60}\text{Ta}_{40}$ 50 nm	 $E_s = 2 \text{ J/cm}^2$ $n = 5$ Number of cycles $N = 30$	~1,5

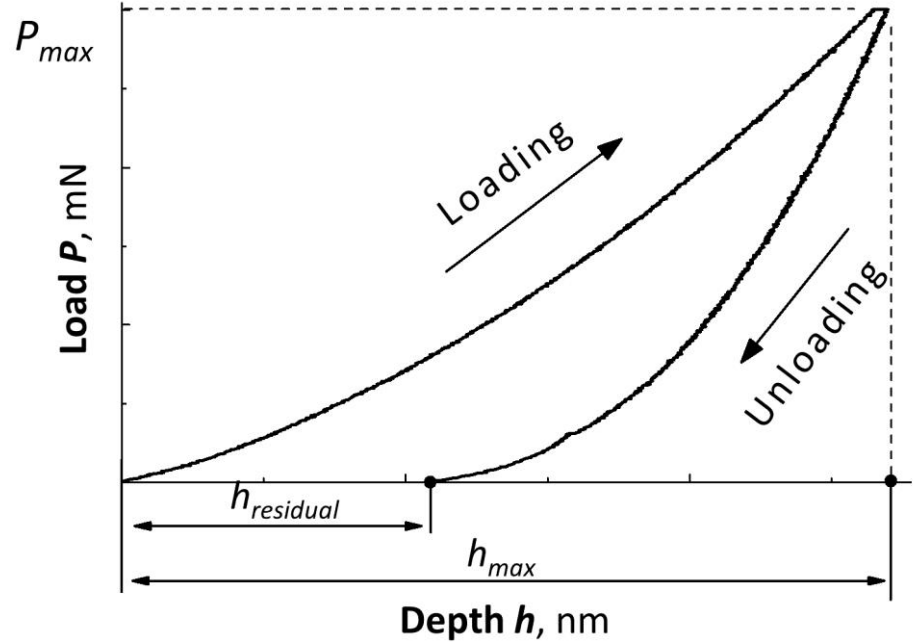


Instrumented indentation

Nanohardness tester NanoTest 600



The example of one indentation cycle



HARDNESS

$$H = \frac{P_{max}}{A}$$

CHARACTERISTIC OF PLASTICITY

$$\delta_H = 1 - 14,3 \times (1 - \nu - 2\nu^2) \frac{HV}{E}$$

ELASTIC MODULUS

$$E = \frac{E_r^2 \times E_i^2 \times (1 - \nu^2)}{E_i^2 - E_r^2 \times (1 - \nu_i^2)}$$

RECOVERY RATIO OF THE INDENT

$$\eta = \frac{h_{inelastic} + h_{elastic}}{h_{max}}$$



Measurement of thin film mechanical properties using nanoindentation / G. M. Pharr, W. C. Oliver // MRS Bulletin. – 1992. – Vol. 17. –P.28–33.
 Plasticity characteristic obtained through hardness measurement / Yu. V. Milman, B. A. Galanov, S. I. Chugunova // Acta Met.Mater. – 1993. –Vol. 41. – P. 2523–2531.
 Shape recovery after nanoindentation of NiTi thin films / W. C. Crone, G. A. Shaw, D. S. Stone, A. D. Johnson, A. B. Ellis // Charlotte. NC. – 2003. – P. 1–6.

Results and discussion

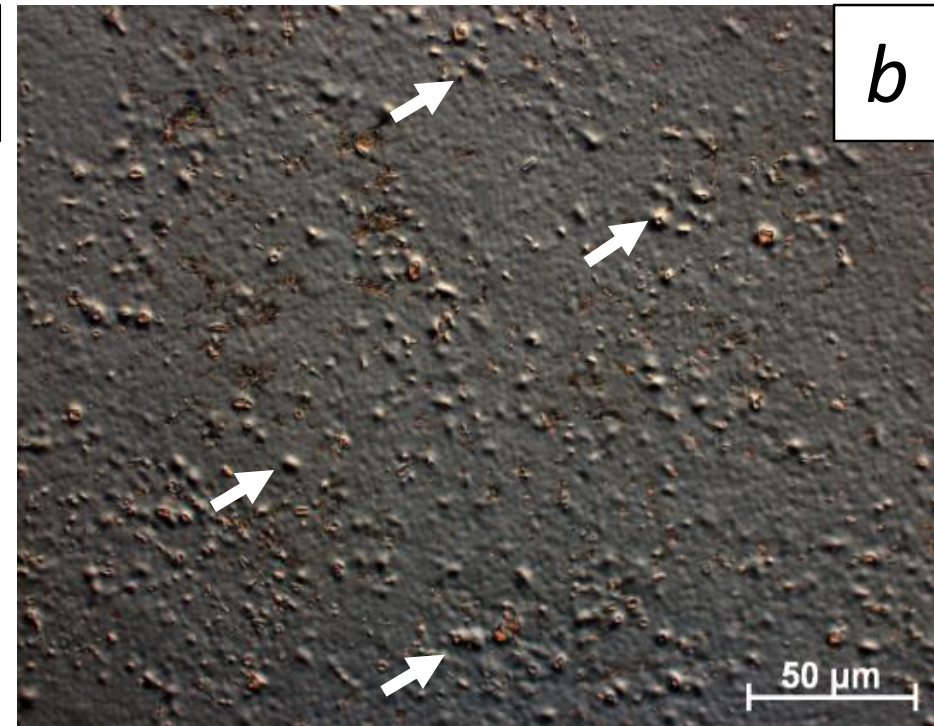
14

Initial NiTi

OPTICAL METALLOGRAPHY



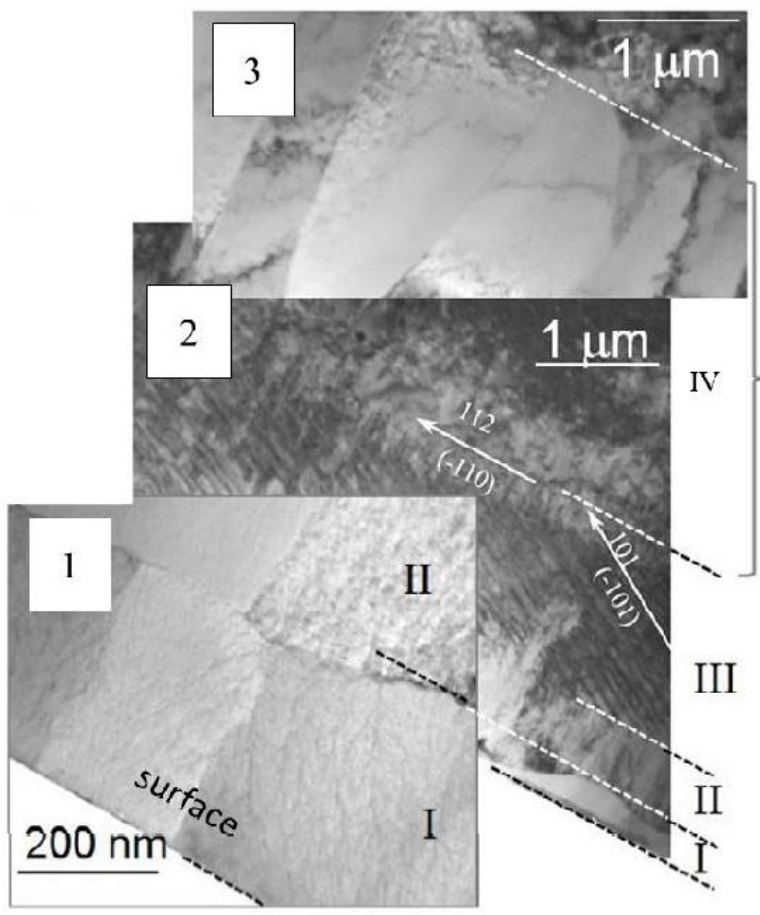
Initial NiTi



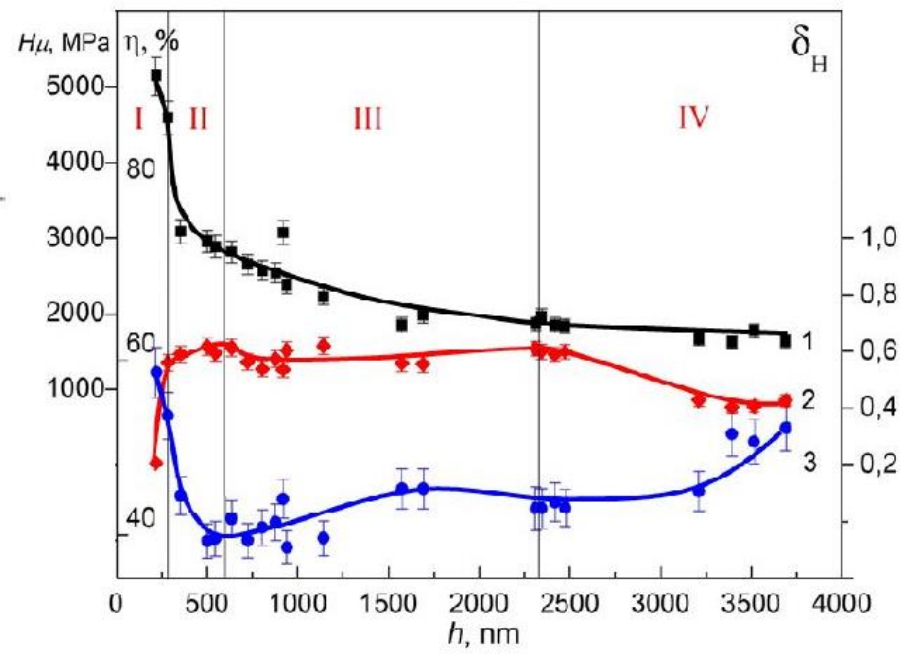
After preliminary surface treatment with out ★ electron beam irradiation

Results and discussion

Electron beam treatment



(a)



(b)

FIGURE 2. Surface microstructure of e-beam treated NiTi (a): 1, 2, 3—images with indication of sublayers I–IV, and dependences of H_{μ} (1), δ_H (2), and η (3) on indentation depth h (b)



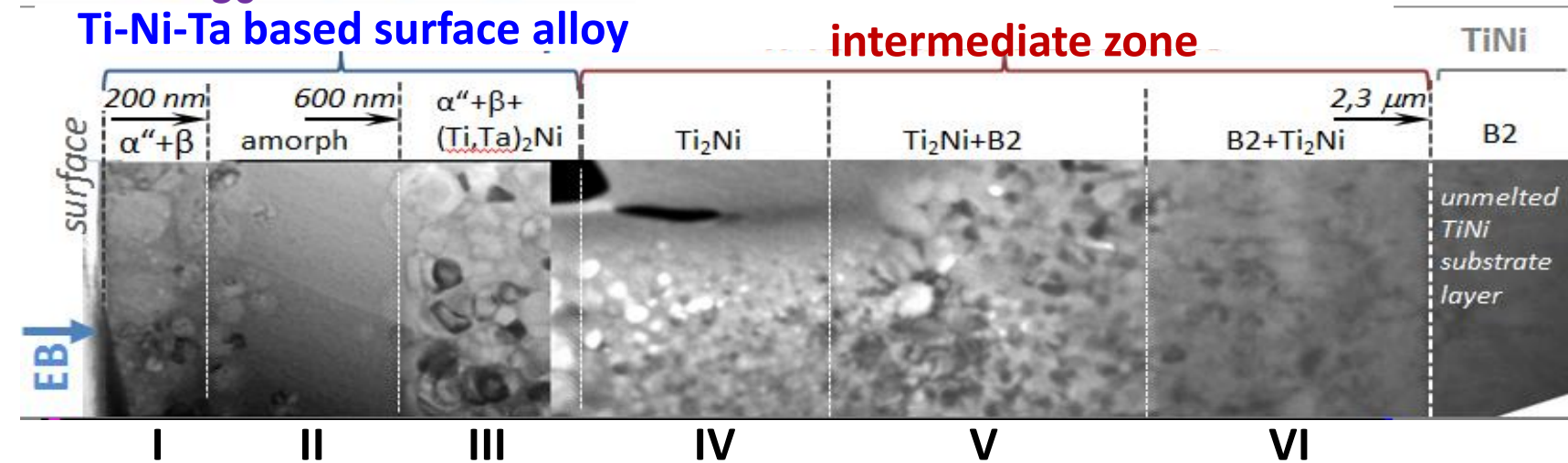
[*] Surface structure and physicomechanical properties of NiTi exposed to electron beam and ion-plasma treatment / S.N. Meisner [et al.] // AIP CP. – 2017. – Vol. 1909. – P. 020134(1-4).

TiNi-Ta₃₀

Ti-Ni-Ta based surface alloy

Ti-Ni-Ta
intermediate zone

TiNi



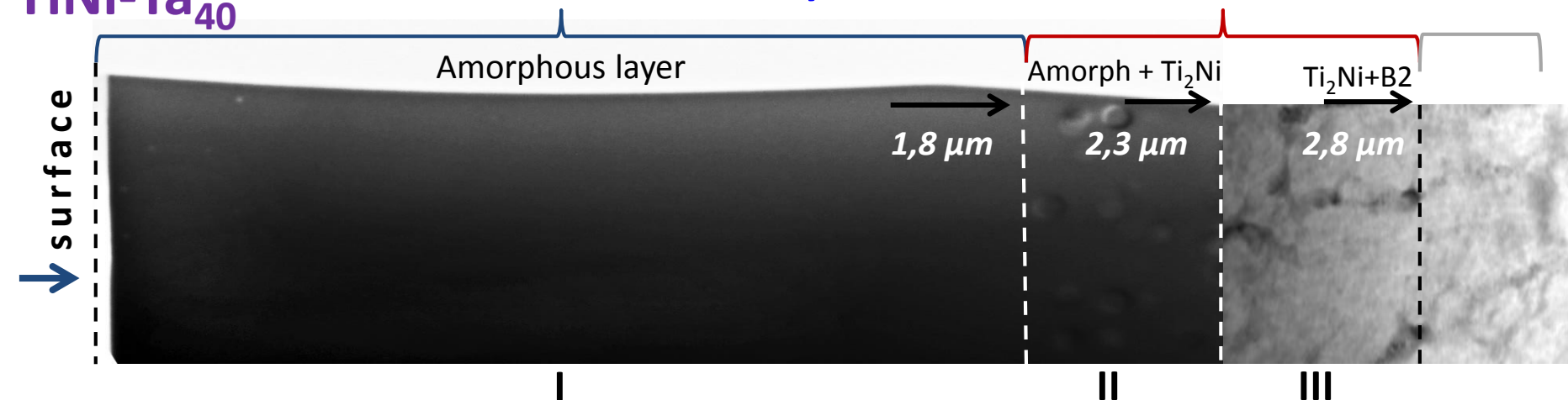
Meisner S. N., Yakovlev E. V., Semin V. O., Meisner L. L., Rotshtein V. P., Neiman A. A., D'yachenko F.A. Mechanical behavior of Ti-Ta based surface alloy fabricated on TiNi SMA by pulsed electron-beam melting of film/substrate system // *Applied Surface Science*. – 2018. – Vol. 437. – P. 217 – 226.

TiNi-Ta₄₀

Ti-Ni-Ta based surface alloy

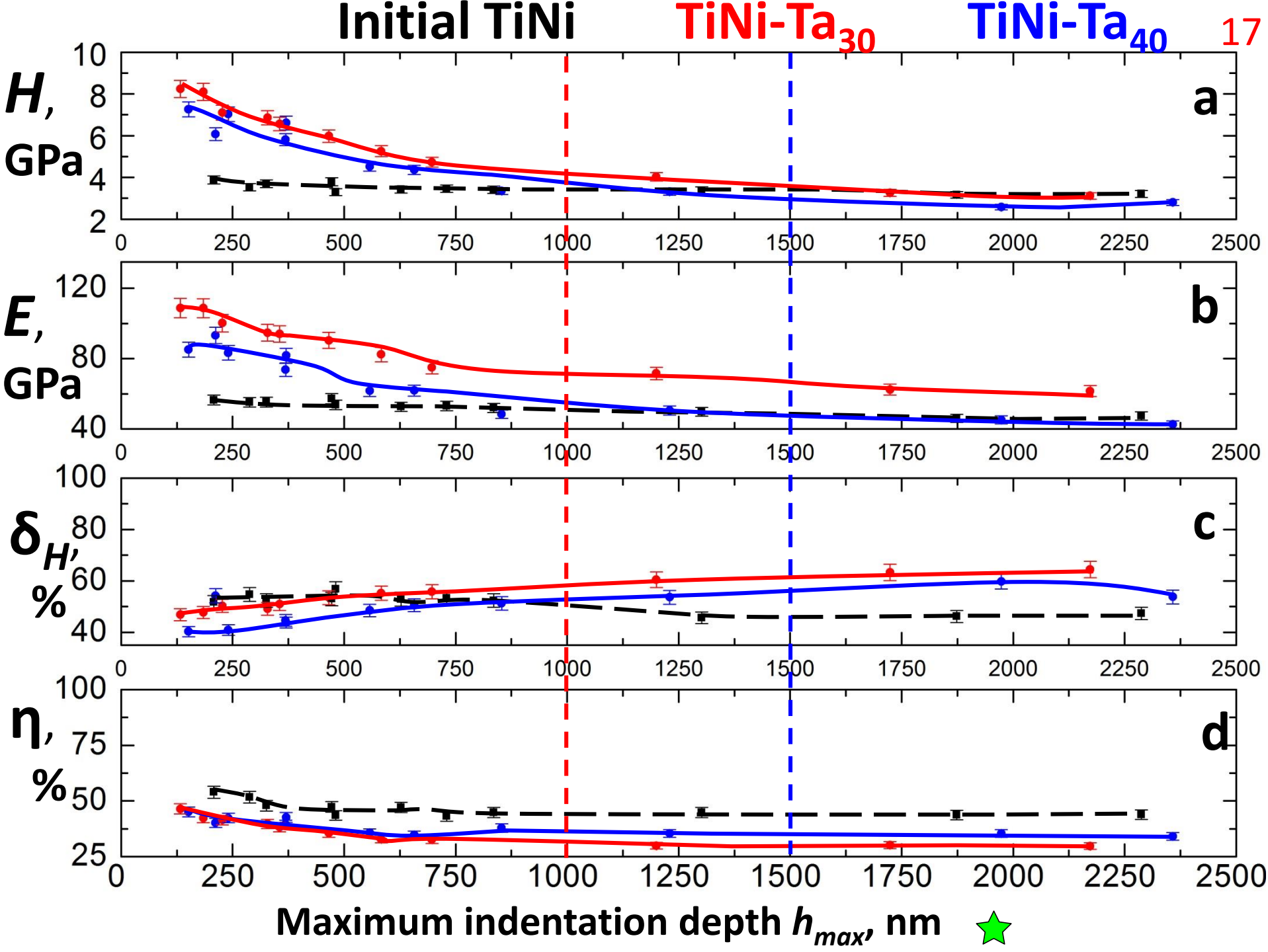
Ti-Ni-Ta
intermediate zone

TiNi



Neiman A., Mukhamedova R., Semin V. Mechanical properties of the TiNi and surface alloy formed by pulsed electron beam treatment // *Materials Research Proceedings*. – 2018. – Vol. 9. – P. 58–62.





Conclusion

The additive thin-film electron beam synthesis of Ti-Ni-Ta surface alloys leads to the formation of the nanocomposite structure with high strength and elastic characteristics of the surface alloys, with a good combination of plasticity properties of the intermediate zone.



References

- *Measurement of thin film mechanical properties using nanoindentation / G. M. Pharr, W. C. Oliver // MRS Bulletin. – 1992. – Vol. 17. –P.28–33.*
- *Plasticity characteristic obtained through hardness measurement / Yu. V. Milman, B. A. Galanov, S. I. Chugunova // Acta Met.Mater. – 1993. –Vol. 41. – P. 2523–2531.*
- *Shape recovery after nanoindentation of NiTi thin films / W. C. Crone, G. A. Shaw, D. S. Stone, A. D. Johnson, A. B. Ellis // Carlotte. NC. – 2003. – P. 1–6.*
- *Surface structure and physicomechanical properties of NiTi exposed to electron beam and ion-plasma treatment / S.N. Meisner [et al.] // AIP CP. – 2017. – Vol. 1909. – P. 020134(1-4).*
- *Meisner S. N., Yakovlev E. V., Semin V. O., Meisner L. L., Rotshtein V. P., Neiman A. A., D'yachenko F.A. Mechanical behavior of Ti-Ta based surface alloy fabricated on TiNi SMA by pulsed electron-beam melting of film/substrate system // Applied Surface Science. – 2018. – Vol. 437. – P. 217 – 226.*
- *Neiman A., Mukhamedova R., Semin V. Mechanical properties of the TiNi and surface alloy formed by pulsed electron beam treatment // Materials Research Proceedings. – 2018. – Vol. 9. – P. 58–62.*



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for conducting electron microscopy investigations,*

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THANK YOU
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