



**iiw**2021  
ON-LINE ASSEMBLY  
AND INTERNATIONAL  
CONFERENCE  
JULY 7/21

74th IIW on-line Assembly and International Conference

C-XVII Brazing, Soldering and Diffusion Bonding

Document number **XVIIIB-0055-21**

# Diffusion bonding of Ti6Al4V to Al<sub>2</sub>O<sub>3</sub> using different interlayer materials



M. Silva Jr.<sup>1,2,3</sup>, A.S. Ramos<sup>4</sup>, M.T. Vieira<sup>4</sup>, S. Simões<sup>2,3</sup>

<sup>1</sup>Department of Mechanical Engineering, Federal University of Amazonas, Manaus 69067-005, Brazil

<sup>2</sup>Department of Metallurgical and Materials Engineering, University of Porto, 4200-465 Porto, Portugal

<sup>3</sup>LAETA/INEGI - Institute of Science and Innovation in Mechanical and Industrial Engineering, 4200-465 Porto, Portugal

<sup>4</sup>University of Coimbra, CEMMPRE, Department of Mechanical Engineering, 3030-788 Coimbra, Portugal

**Joining of Ti alloys to ceramic materials** can contribute to overcome the limitations that titanium alloys present when the application requires operating temperatures above 550 °C.

However,

Processing of high quality and high strength dissimilar joints between Ti alloys and ceramic materials constitutes a major challenge!

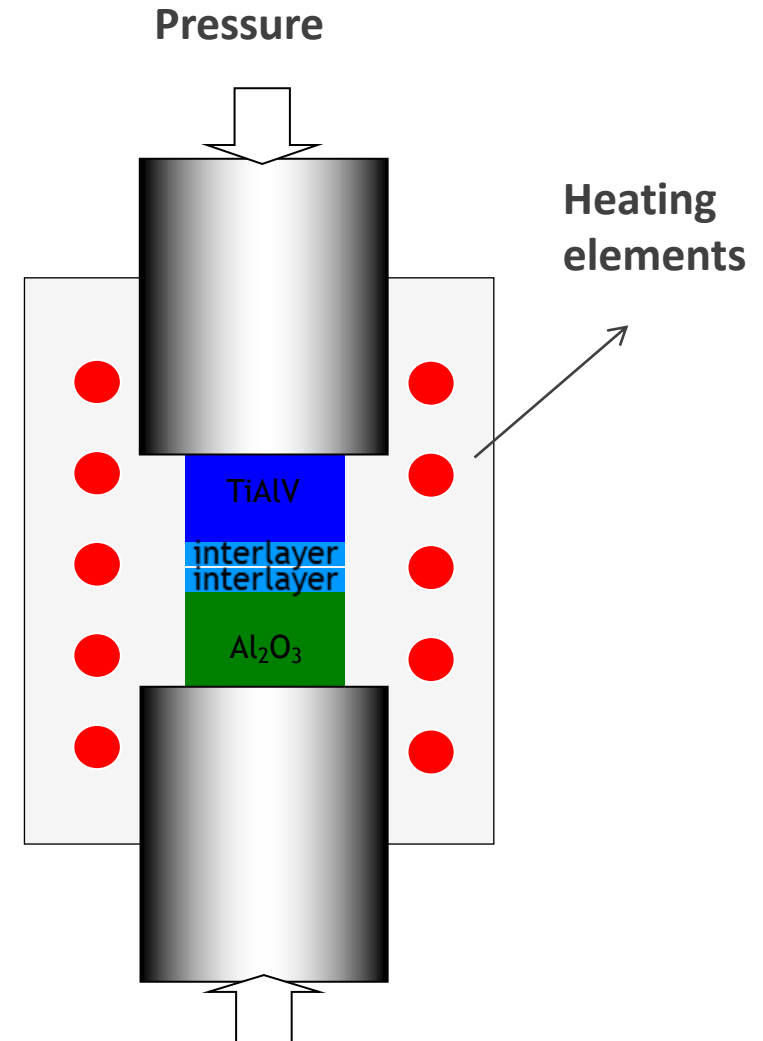
- **Brazing** and **diffusion bonding** are the most reported technologies for metal to ceramic joining.
- Reactive multilayer thin films have been successfully used as filler material for similar and dissimilar joining of metals by diffusion bonding – **Reaction-Assisted Diffusion Bonding**.

The aim of the present work is to develop strategies for the successful joining of Ti6Al4V to Al<sub>2</sub>O<sub>3</sub> by diffusion bonding

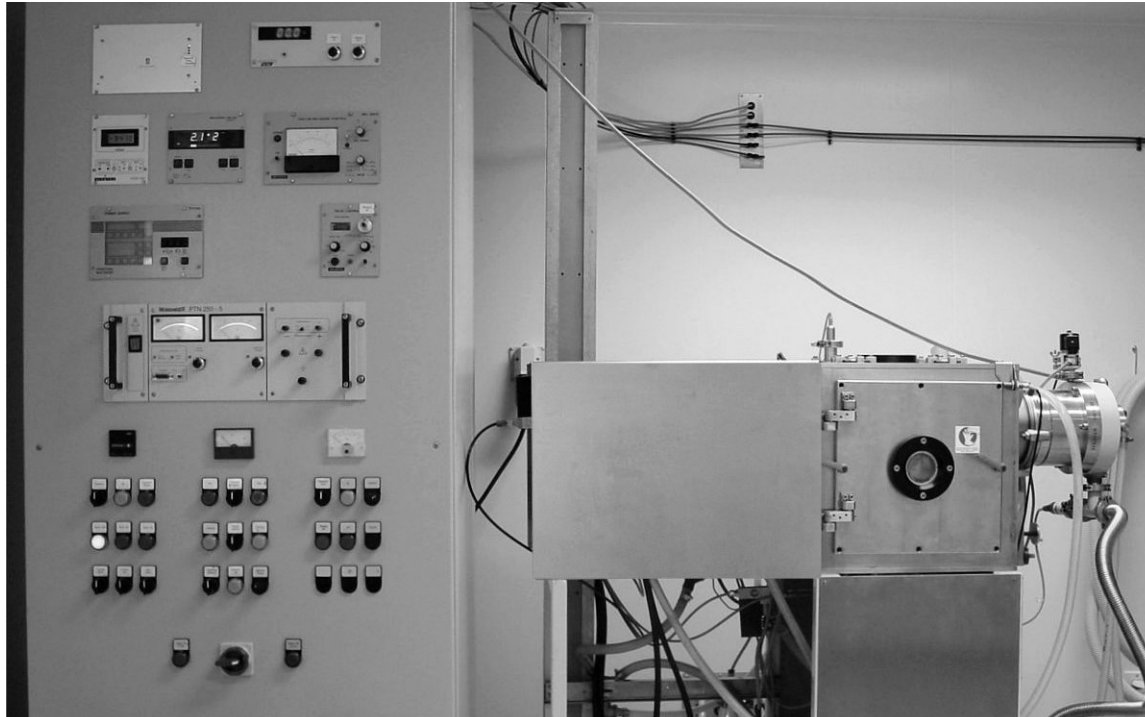
Interlayer materials selected for diffusion bonding Ti6Al4V to  $\text{Al}_2\text{O}_3$ :

- i) Ni/Ti reactive multilayer thin films
- ii) Ti monolithic thin films
- iii) Ti thin foils ( $5\ \mu\text{m}$  thick)

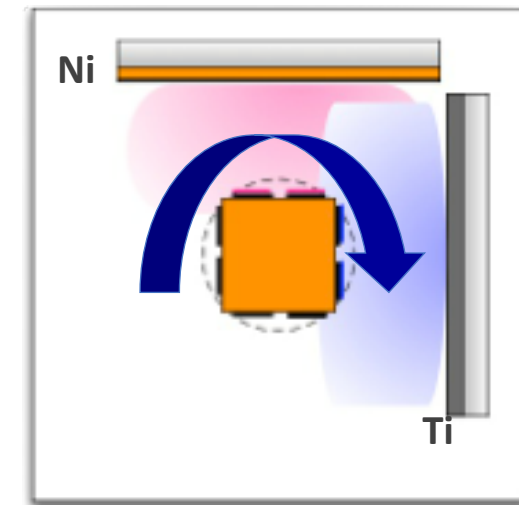
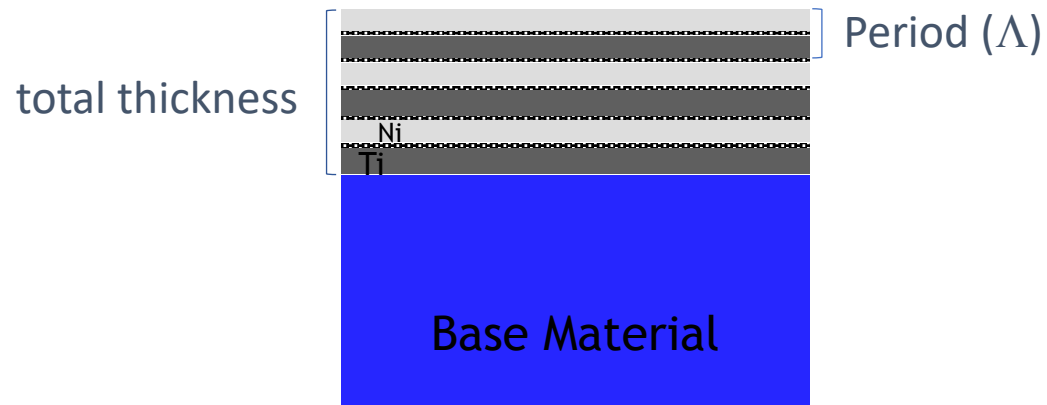
The objective is to reduce the diffusion bonding temperature, time and/or pressure (as successfully done with metallic materials)



Interlayers i) and ii) deposited onto the base materials by *magnetron sputtering*

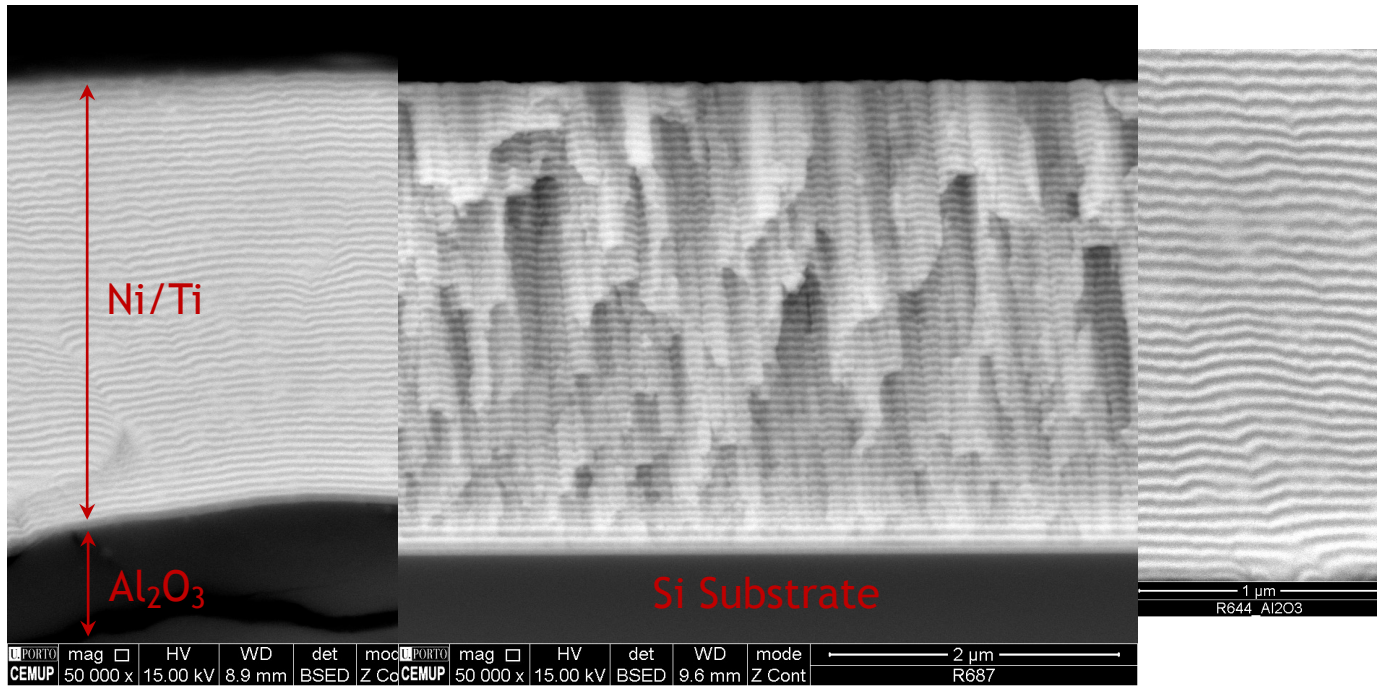


- DC magnetron sputtering
- High purity Ti (and Ni) targets
- Base pressure  $< 5 \times 10^{-4}$  Pa
- Substrates cleaned by Heating/Etching
- Deposition pressure = 0.4 Pa
- Substrate bias = -40 V (-70 for Ti films)
- Total thickness = 1.0 - 3.0  $\mu\text{m}$
- $\Lambda \approx 12, 25$  and 50 nm

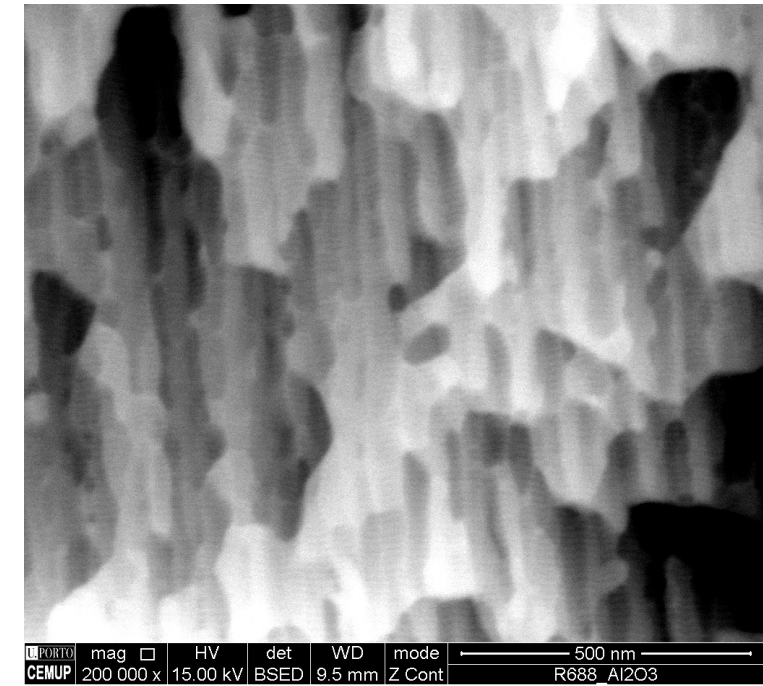


Ni/Ti multilayer thin films with nanometric modulation period ( $\Lambda$ ) deposited onto  $\text{Al}_2\text{O}_3$

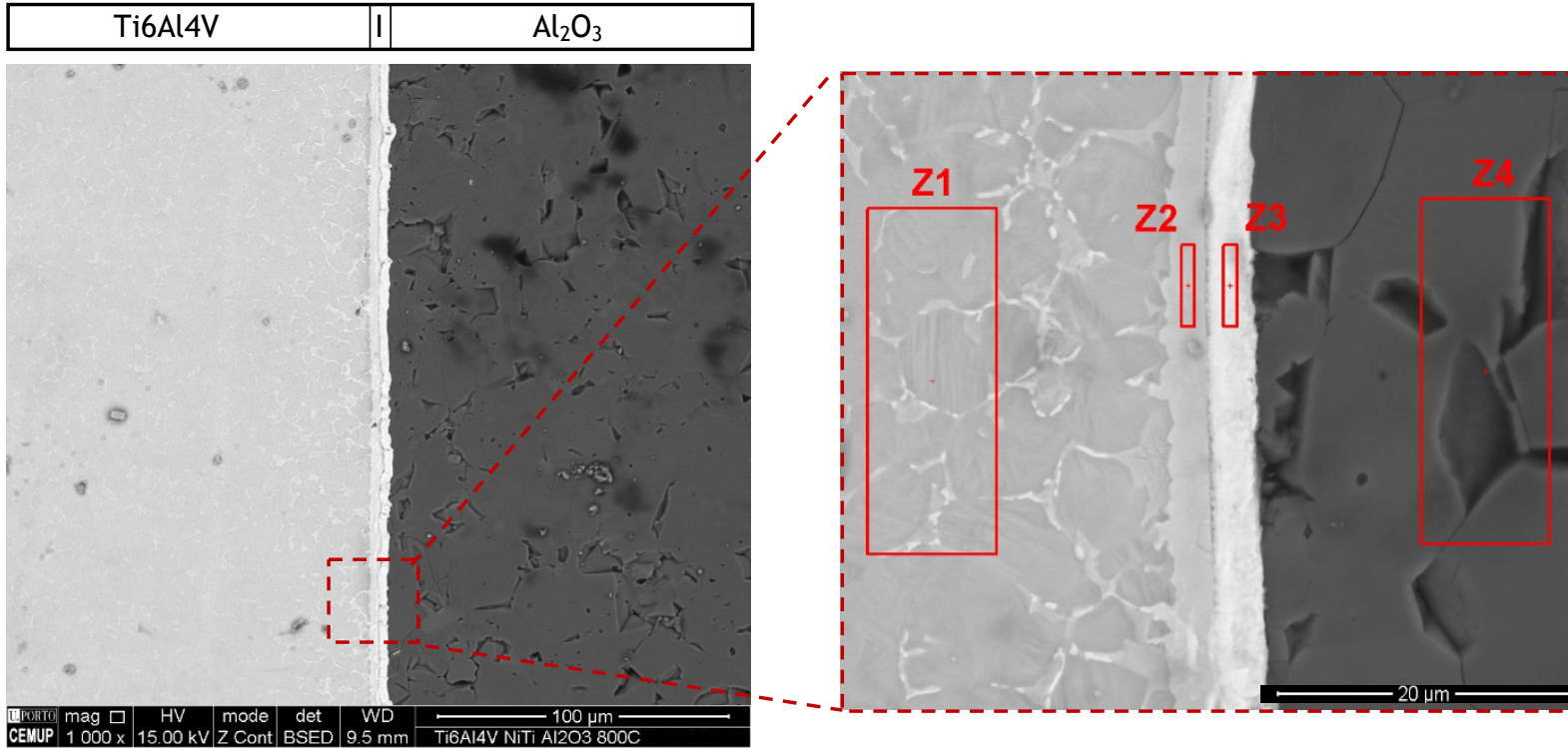
$\Lambda \approx 50 \text{ nm}$



$\Lambda \approx 12 \text{ nm}$



Ni/Ti  $\Lambda = 50$  nm @ T = 800°C (50 MPa / 60 min)



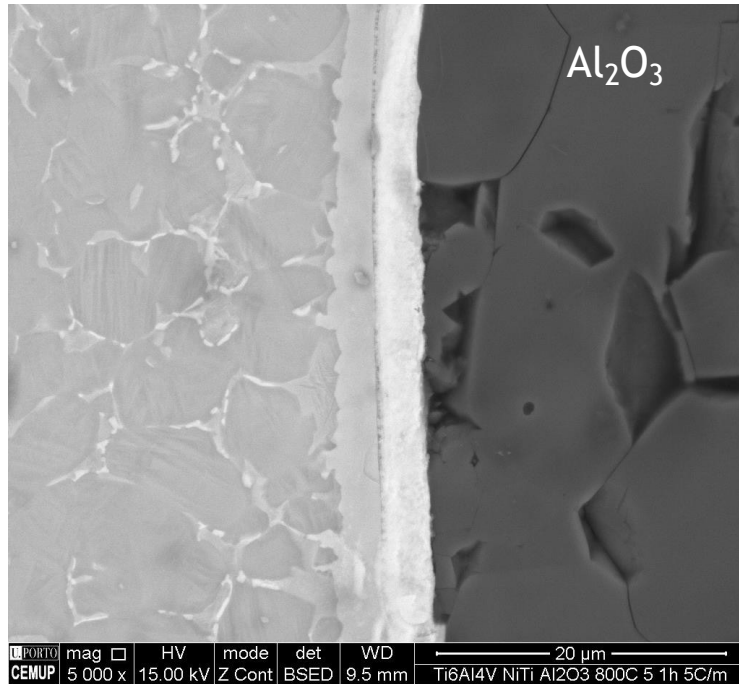
Heating rate of 10°C/min  
Cooling rate of 5°C/min up to 500°C, followed by 3°C/min down to RT

	Ti (at. %)	Al (at. %)	Ni (at. %)	V (at. %)	O (at. %)	Possible Phases
Z1	86.0	9.9	1.3	2.8	*	$\alpha$ -Ti + $\beta$ -Ti
Z2	64.3	1.9	32.1	1.7	*	NiTi <sub>2</sub>
Z3	46.5	—	50.4	3.1	*	NiTi
Z4	—	43.8	—	—	56.2	Al <sub>2</sub> O <sub>3</sub>

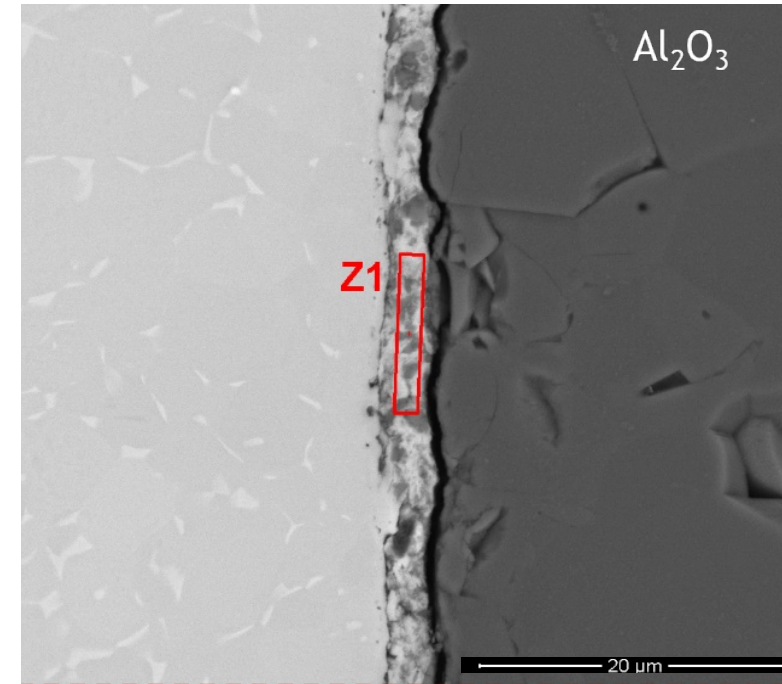
\*Not quantified

800°C / 50 MPa / 60 min

With interlayer



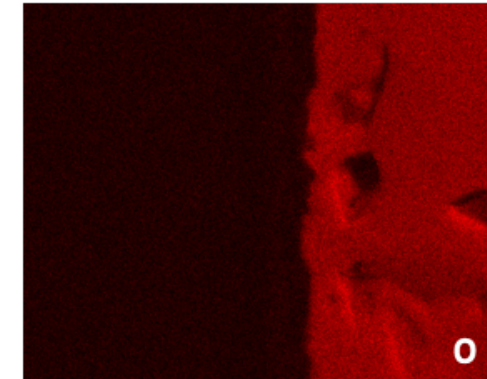
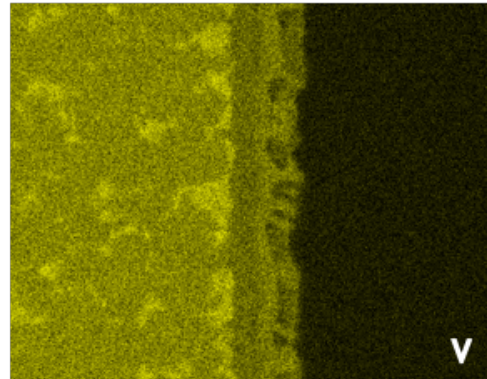
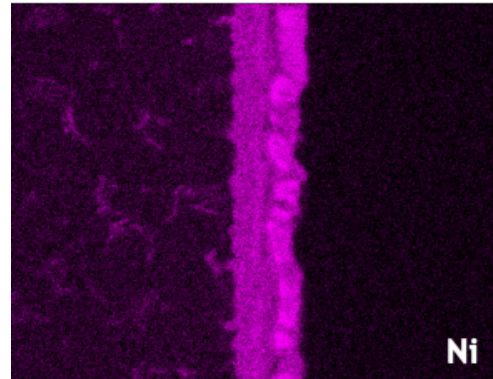
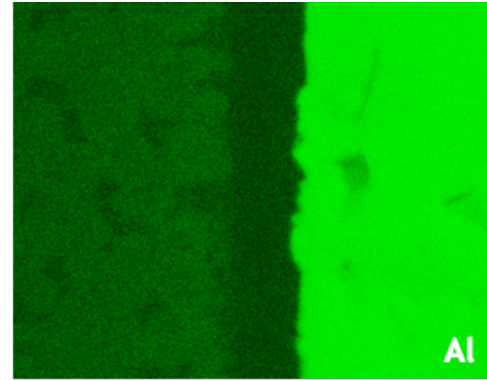
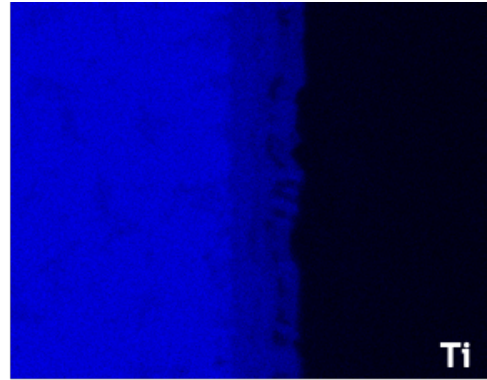
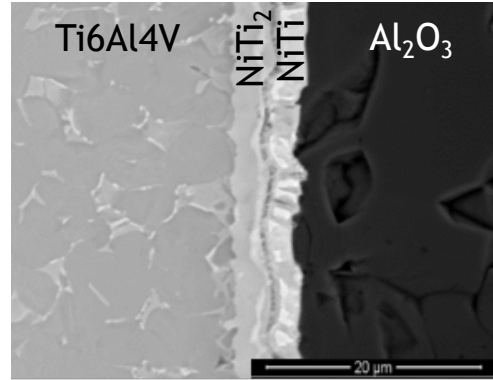
Without interlayer



Zone Z1

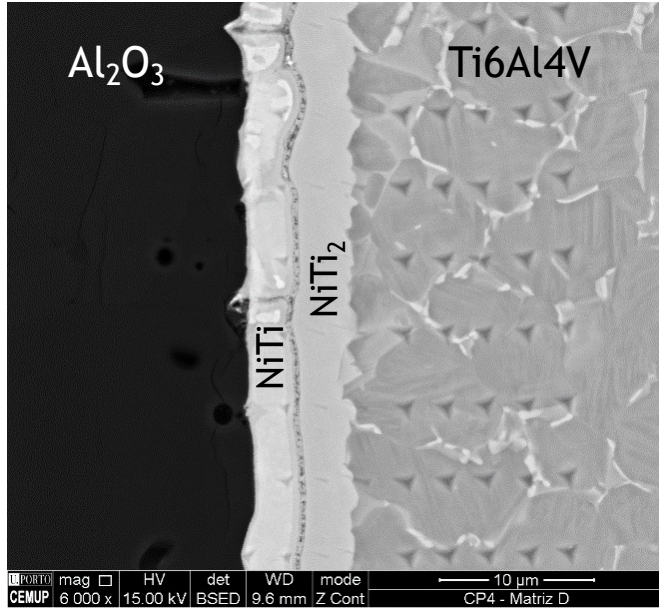
48.5 at.% Ti, 39.3 at.% O, 11.3 at.% Al, 0.9 at.% V

Ni/Ti  $\Lambda = 50$  nm @ T = 800°C (50 MPa / 60 min)





Ni/Ti  $\Lambda = 50$  nm @ T = 800°C (50 MPa / 60 min)

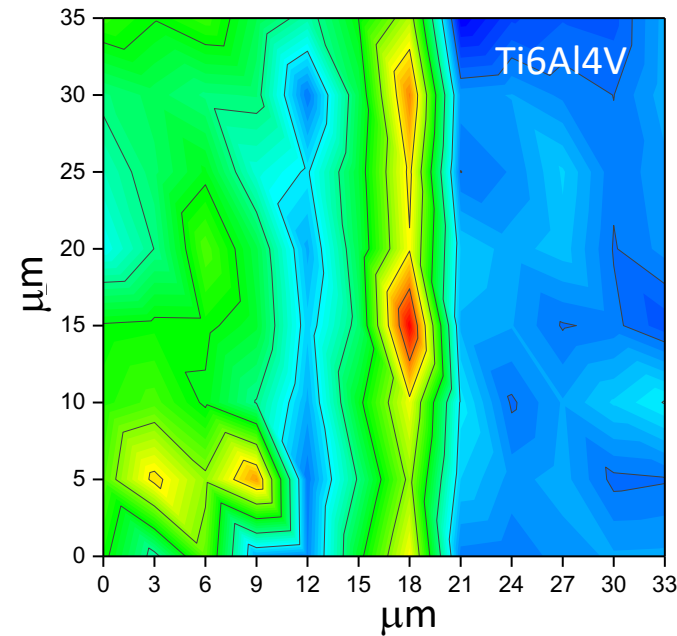
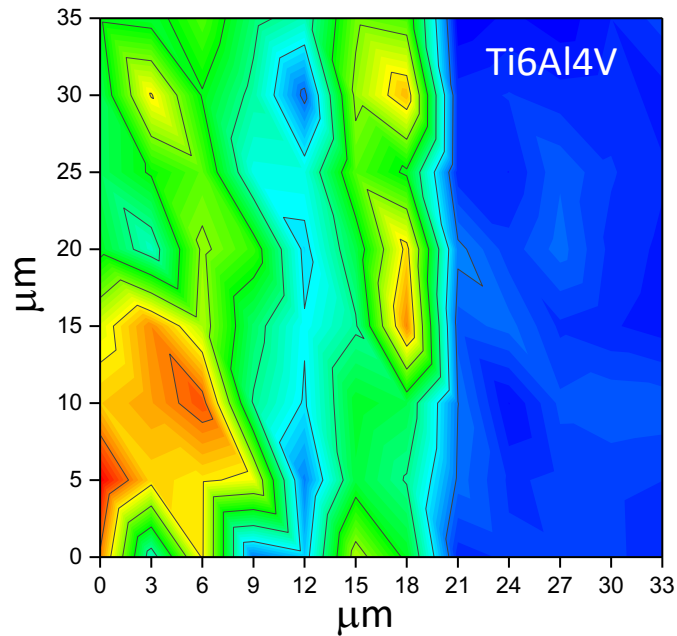


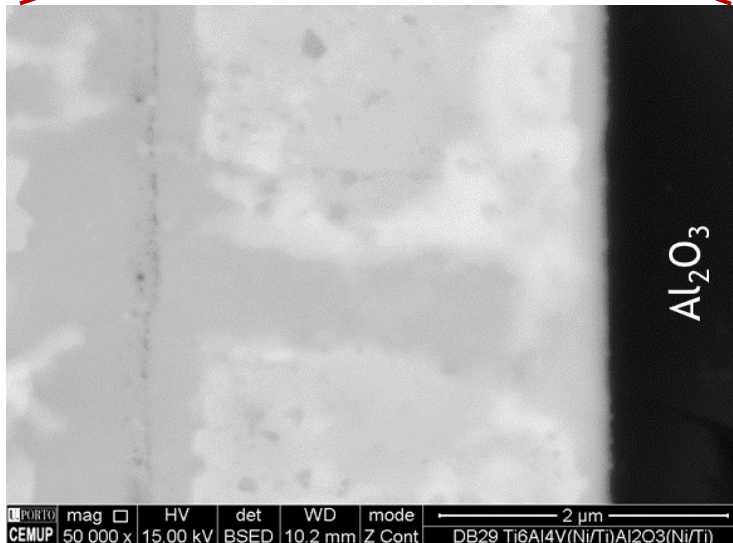
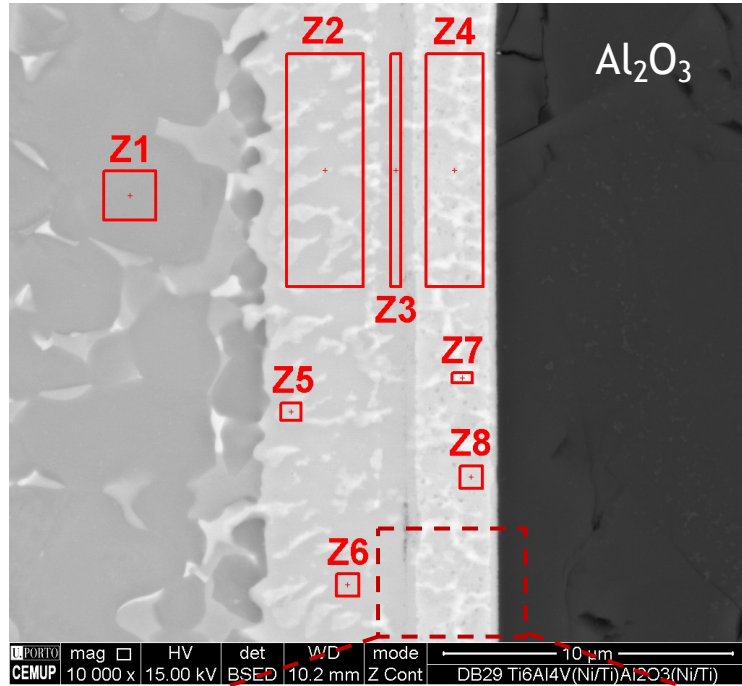
### Nanoindentation

Matrix 8x12 L<sub>max</sub> = 5 mN

Distance between columns = 3 μm

Distance between rows = 5 μm



Ni/Ti  $\Lambda = 50$  nm @ T = 800°C (5 MPa / 60 min)

	Ti (at. %)	Al (at. %)	Ni (at. %)	V (at. %)	Possible Phases
Z1	87.1	10.9	—	2.0	$\alpha$ -Ti
Z2	62.8	3.1	32.2	2.0	NiTi <sub>2</sub>
Z3	60.2	0.68	36.1	3.0	NiTi <sub>2</sub>
Z4	47.9	3.3	45.3	3.6	NiTi
Z5	61.6	3.0	34.0	1.3	NiTi <sub>2</sub>
Z6	62.0	1.6	33.9	2.5	NiTi <sub>2</sub>
Z7	43.9	2.0	50.3	3.8	NiTi
Z8	46.4	1.2	49.0	3.4	NiTi

**Thicker interface on the TiAlV side ???**

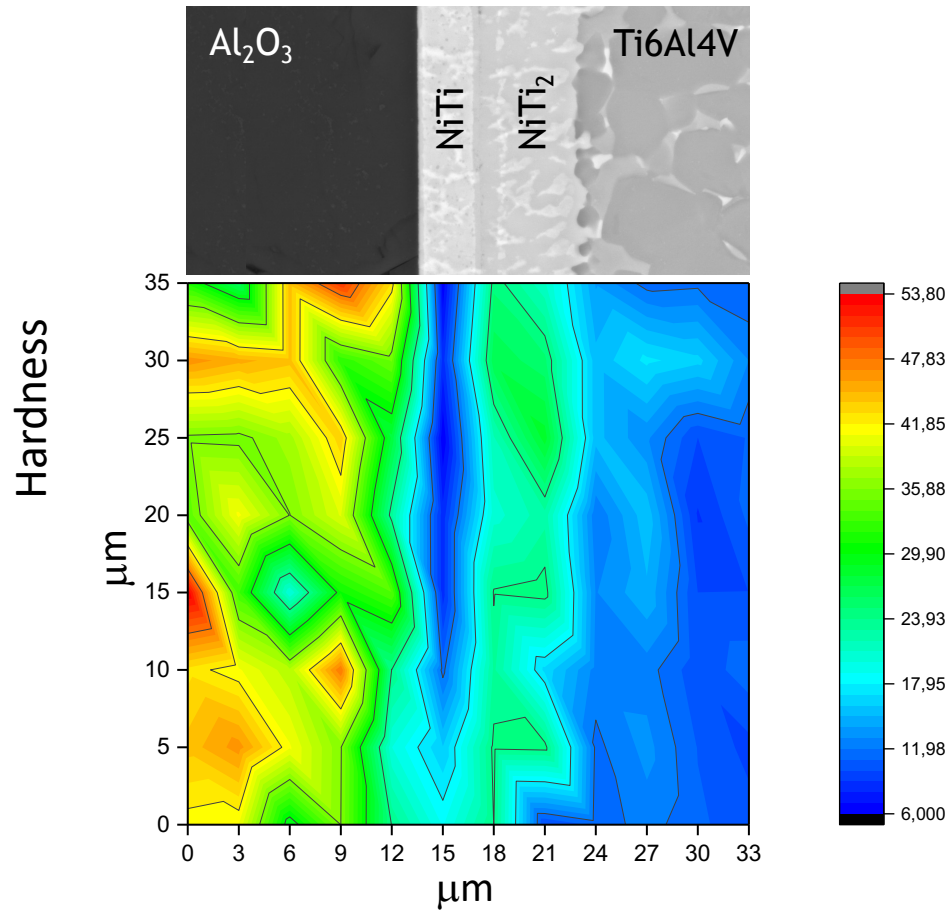
Ni/Ti  $\Lambda = 50$  nm @ T = 800°C (5 MPa / 60 min)

### Nanoindentation

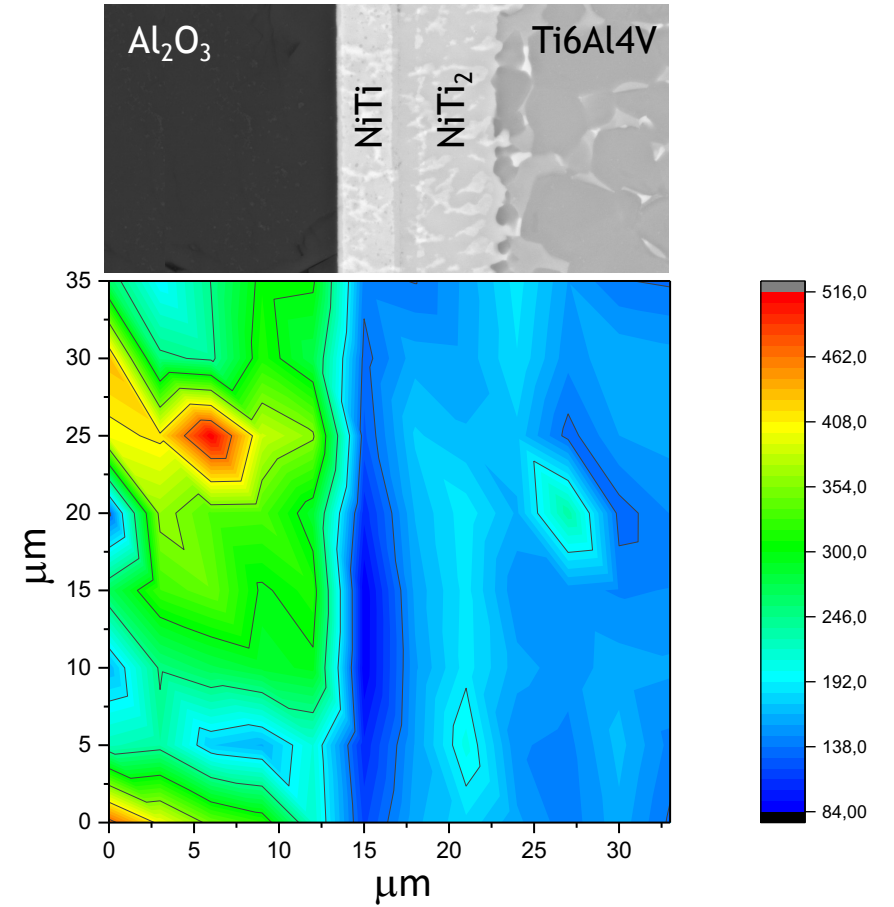
Matrix 8x12  $L_{max} = 5$  mN

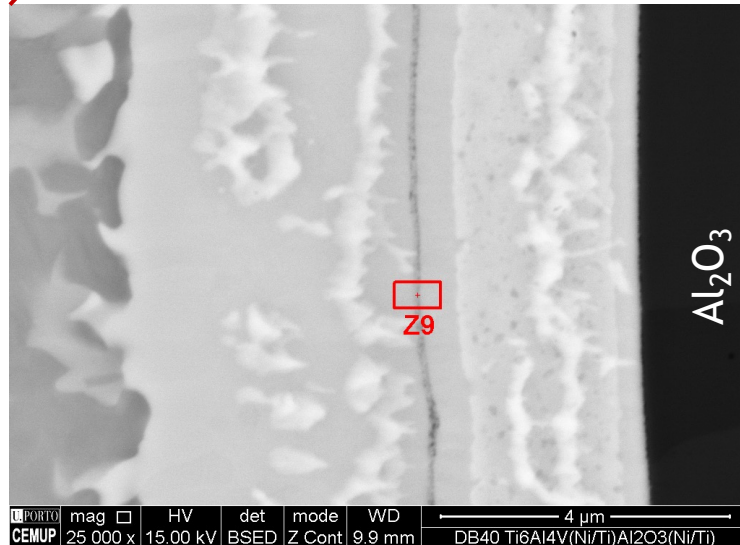
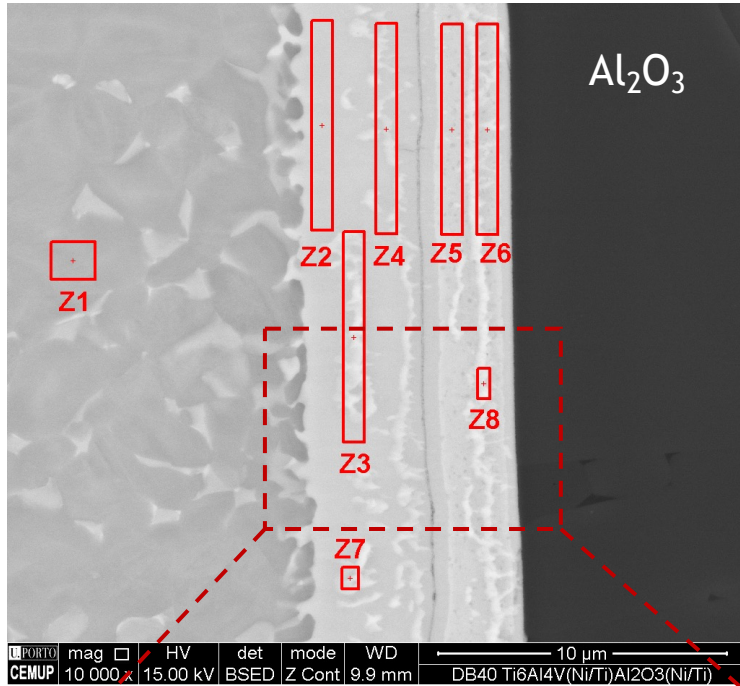
Distance between columns = 3  $\mu$ m

Distance between rows = 5  $\mu$ m



### Reduced Young modulus



Ni/Ti  $\Lambda = 25$  nm @ T = 800°C (5 MPa / 60 min)

	Ti (at. %)	Al (at. %)	Ni (at. %)	V (at. %)	Possible Phases
Z1	86.0	11.0	—	3.0	$\alpha$ -Ti
Z2	65.0	4.4	28.7	1.8	(Ti) + NiTi <sub>2</sub>
Z3	63.3	2.8	31.4	2.4	NiTi <sub>2</sub>
Z4	65.2	1.7	30.2	2.9	(Ti) + NiTi <sub>2</sub>
Z5	53.9	1.0	41.0	4.1	NiTi + (NiTi <sub>2</sub> )
Z6	50.7	1.5	44.0	3.8	NiTi + (NiTi <sub>2</sub> )
Z7	63.5	3.7	30.8	2.0	NiTi
Z8	48.7	0.85	47.4	3.1	NiTi
Z9	68.1	—	29.0	3.0	Ti + NiTi <sub>2</sub>



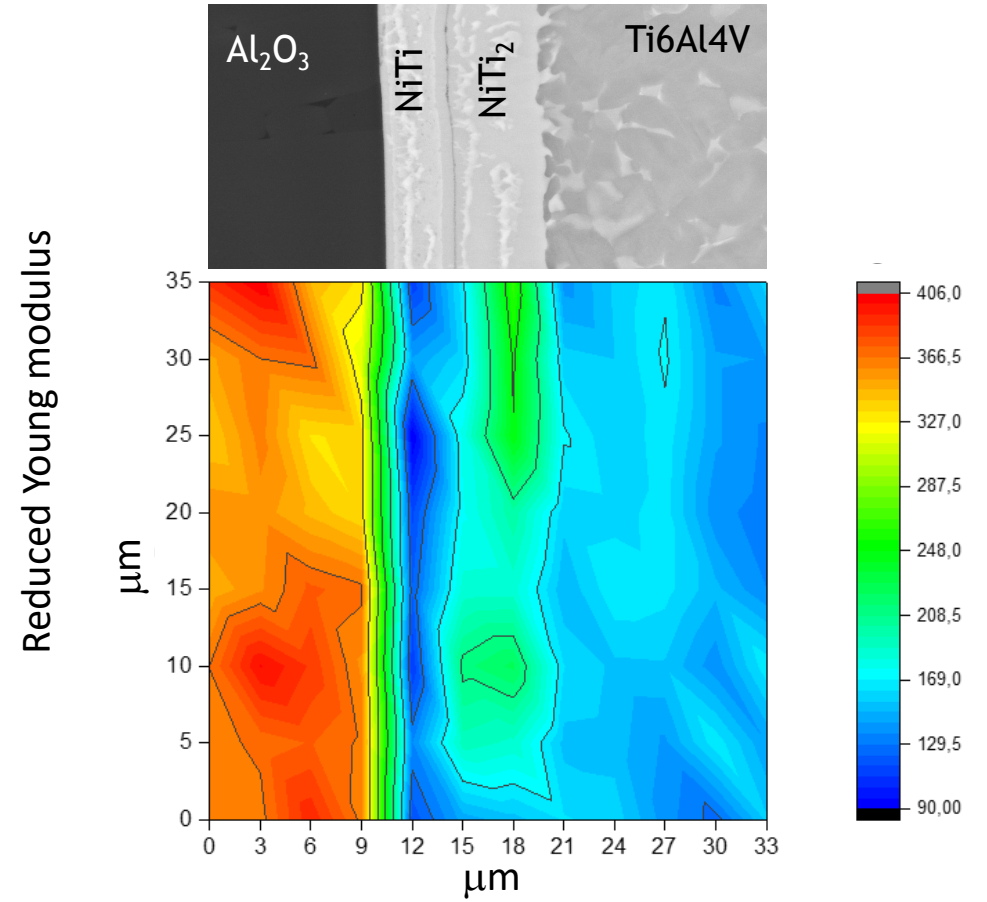
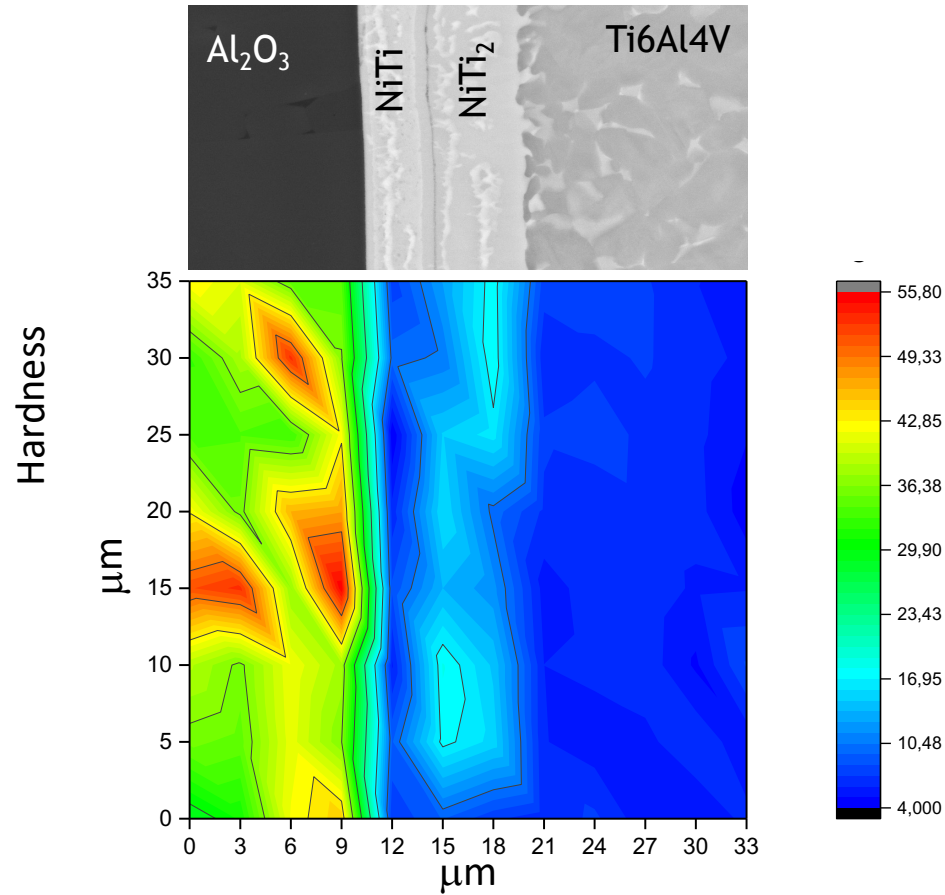
Ni/Ti  $\Lambda = 25$  nm @ T = 800°C (5 MPa / 60 min)

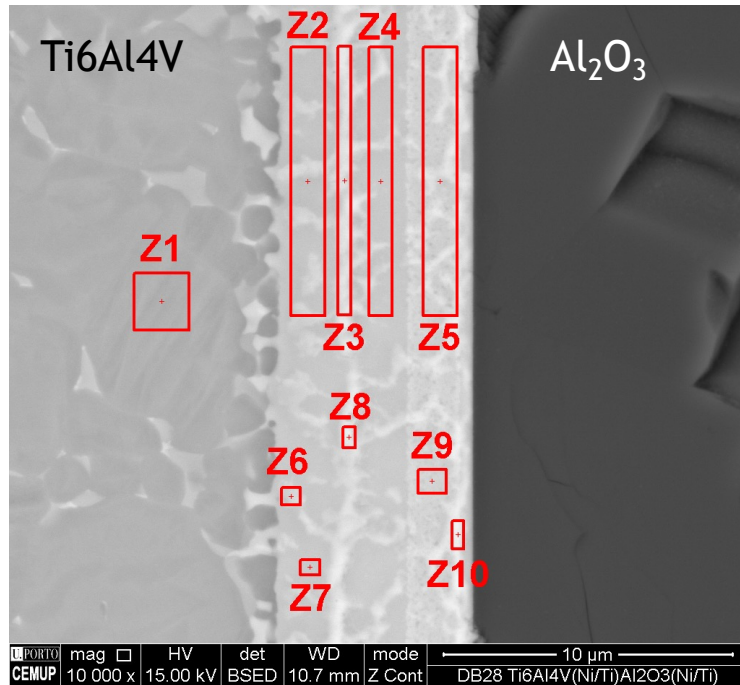
### Nanoindentation

Matrix 8x12  $L_{max} = 5$  mN

Distance between columns = 3  $\mu$ m

Distance between rows = 5  $\mu$ m

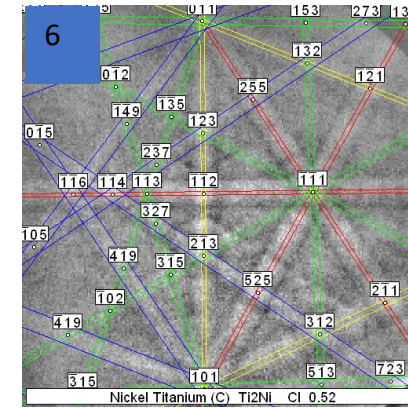
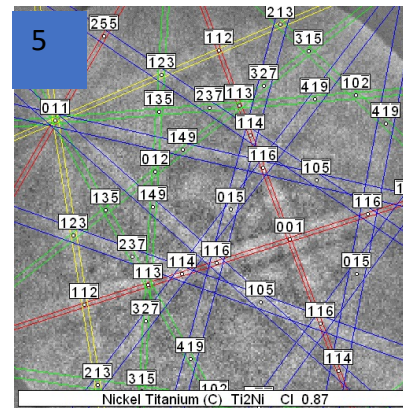
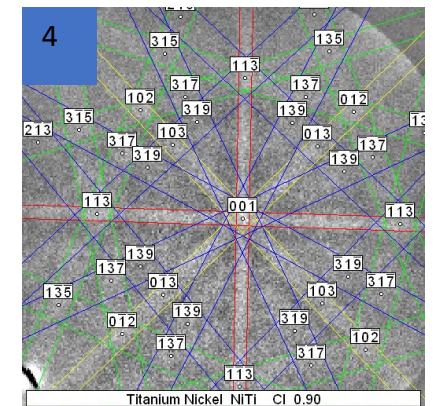
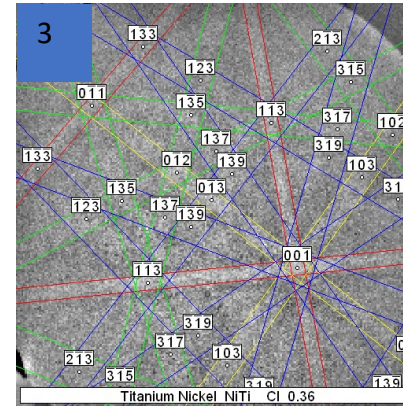
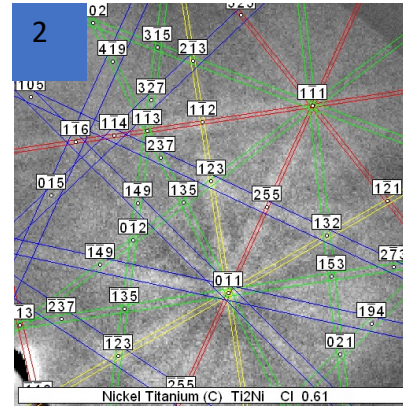
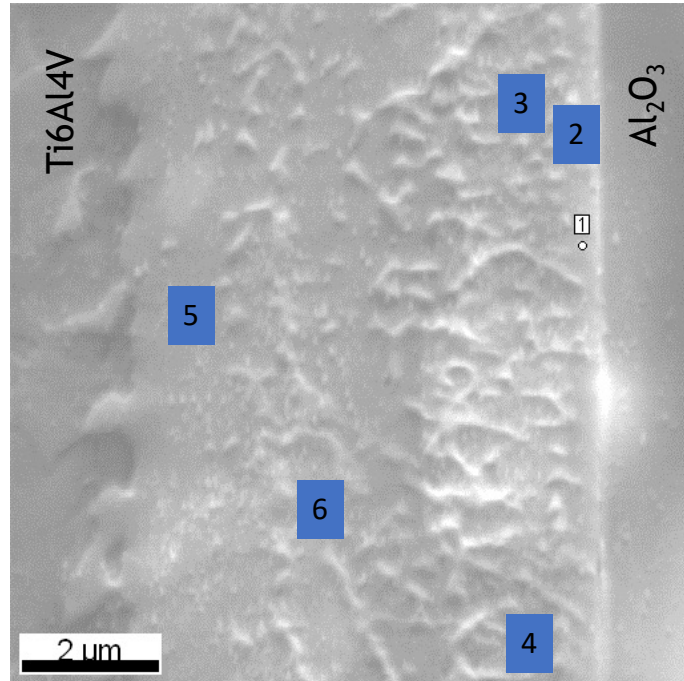


Ni/Ti  $\Lambda = 12$  nm @ T = 800°C (5 MPa / 60 min)Al<sub>2</sub>O<sub>3</sub>

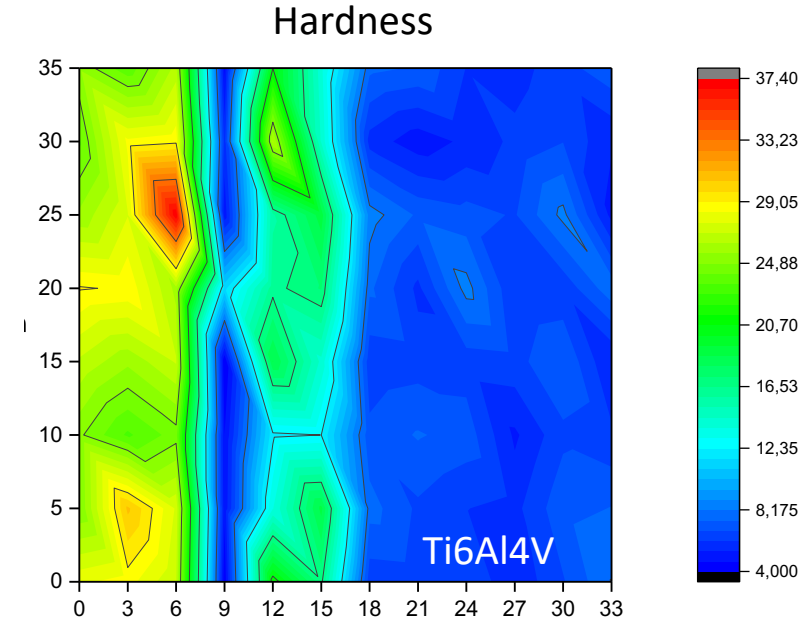
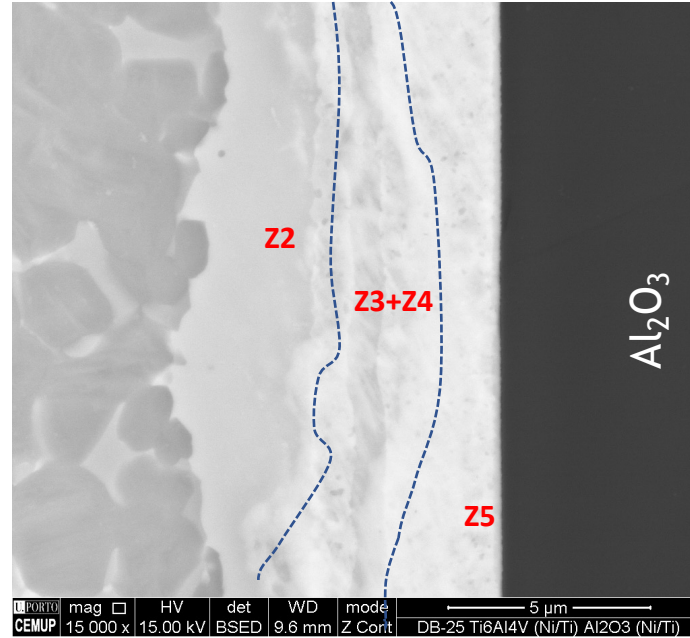
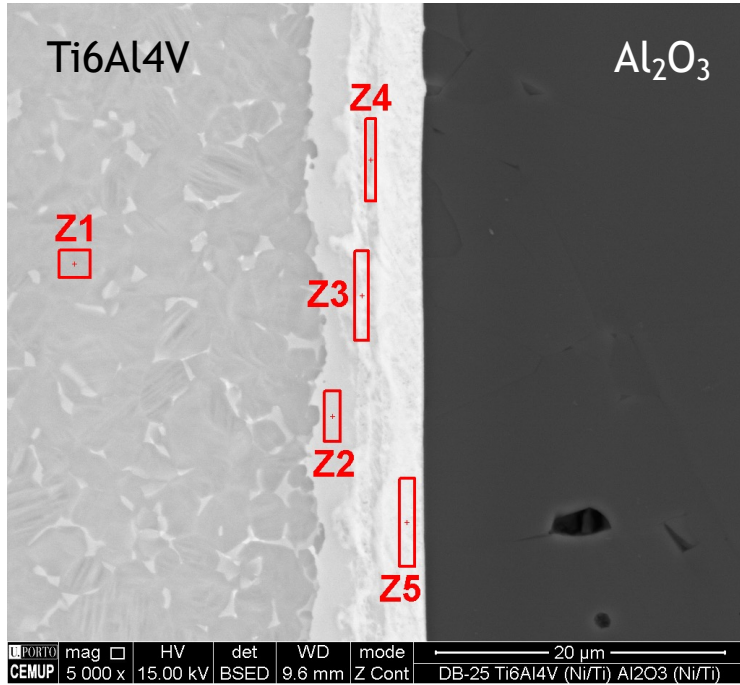
	Ti (at. %)	Al (at. %)	Ni (at. %)	V (at. %)	Possible Phases
Z1	88.6	9.7	–	1.7	α-Ti
Z2	64.9	3.7	29.2	2.2	(Ti) + NiTi <sub>2</sub>
Z3	60.8	1.5	35.1	2.5	(Ti) + NiTi <sub>2</sub>
Z4	61.1	1.1	34.9	3.0	(Ti) + NiTi <sub>2</sub>
Z5	50.2	1.4	45.0	3.3	NiTi + (NiTi <sub>2</sub> )
Z6	60.6	3.2	34.7	1.5	NiTi <sub>2</sub>
Z7	62.6	2.7	33.3	1.3	NiTi <sub>2</sub>
Z8	58.4	1.6	37.6	2.5	NiTi + NiTi <sub>2</sub>
Z9	49.4	0.81	46.8	3.0	NiTi
Z10	44.3	3.5	49.1	3.2	NiTi

Ni/Ti  $\Lambda = 12$  nm @ T = 800°C (5 MPa / 60 min)

EBSD



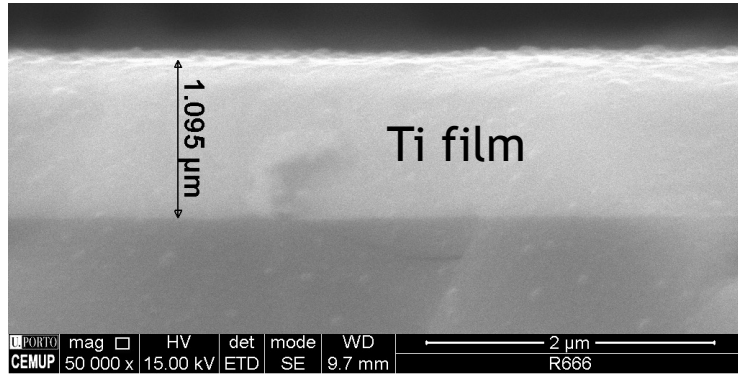
Ni/Ti  $\Lambda = 50$  nm @ T = 750°C (5 MPa / 60 min)



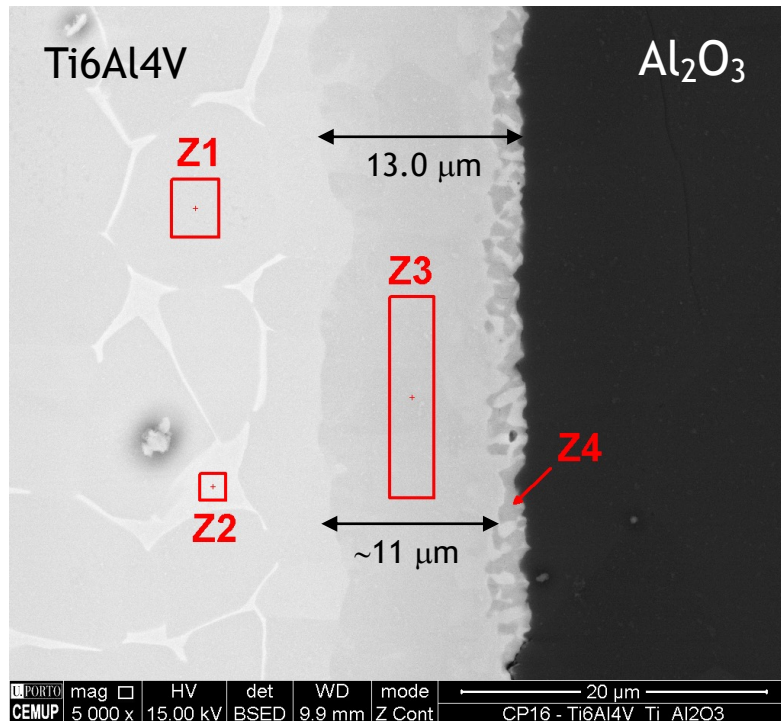
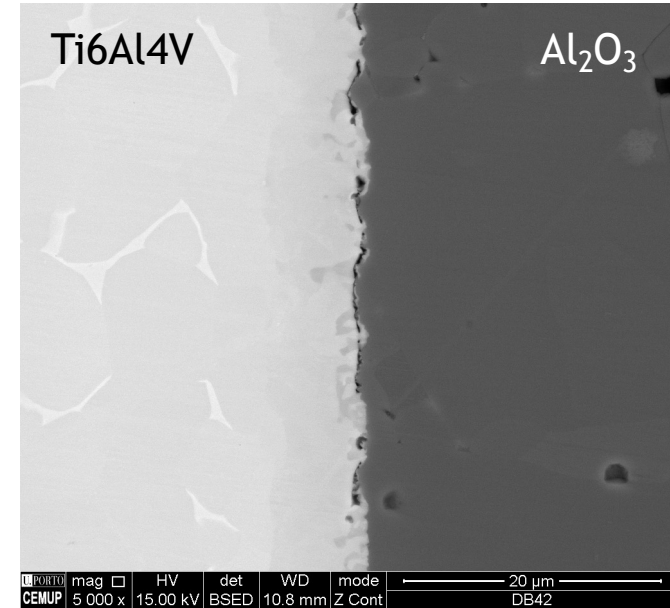
	Ti (at. %)	Al (at. %)	Ni (at. %)	V (at. %)	Possible Phases
Z1	88.1	10.1	—	1.8	α-Ti
Z2	63.7	2.9	31.7	1.7	(Ti) + NiTi <sub>2</sub>
Z3	50.5	0.6	45.5	3.4	NiTi + NiTi <sub>2</sub>
Z4	53.0	0.6	42.6	3.8	NiTi + NiTi <sub>2</sub>
Z5	45.4	0.7	50.6	3.3	NiTi



Ti 1 μm @ T = 1000°C (Contact/ 60 min)



Without interlayer



	Ti (at. %)	Al (at. %)	V (at. %)	Possible Phases
Z1	88.3	11.7	—	α-Ti
Z2	77.2	8.9	16.0	β-Ti
Z3	74.5	25.5	—	Ti <sub>3</sub> Al
Z4	57.6	42.4	—	TiAl + Ti <sub>3</sub> Al

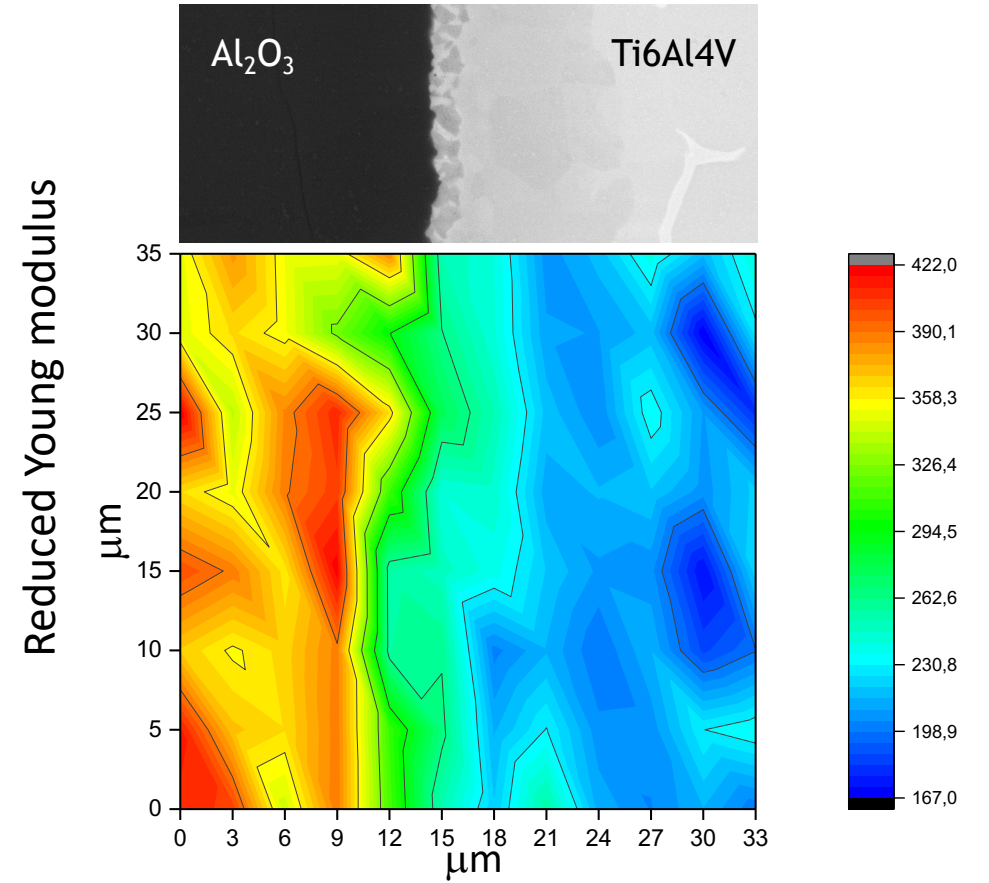
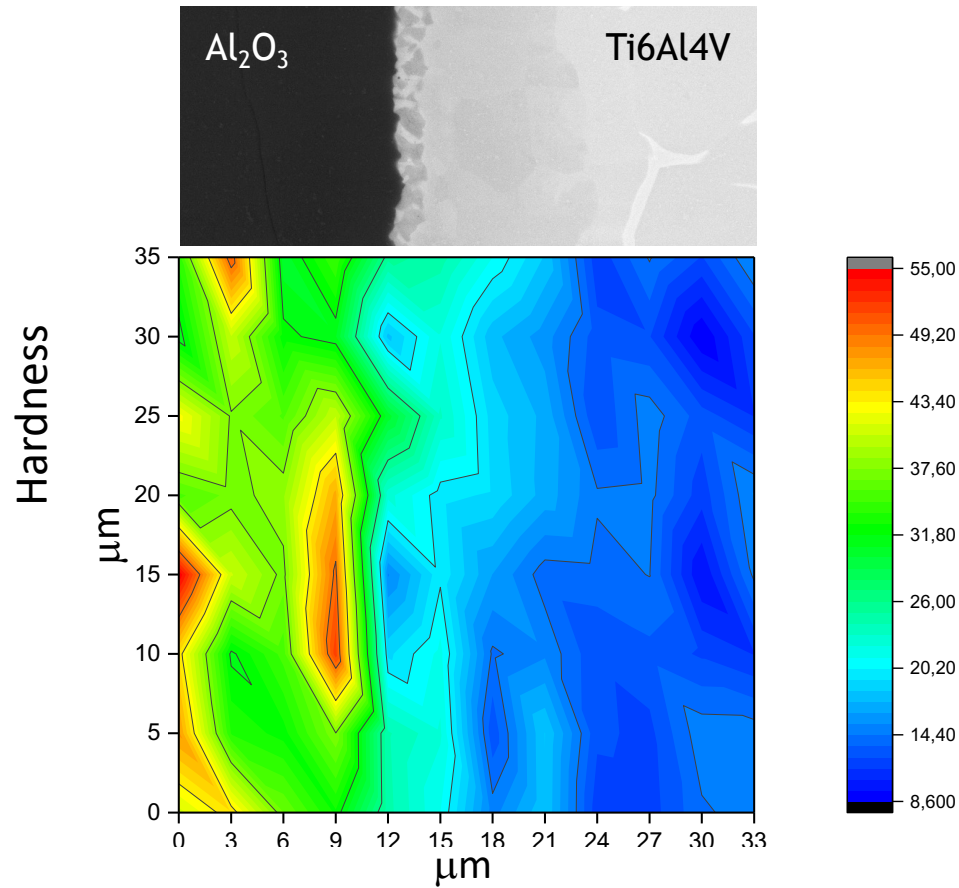
Ti thin film (1 μm) @ T = 1000°C (Contact/ 60 min)

### Nanoindentation

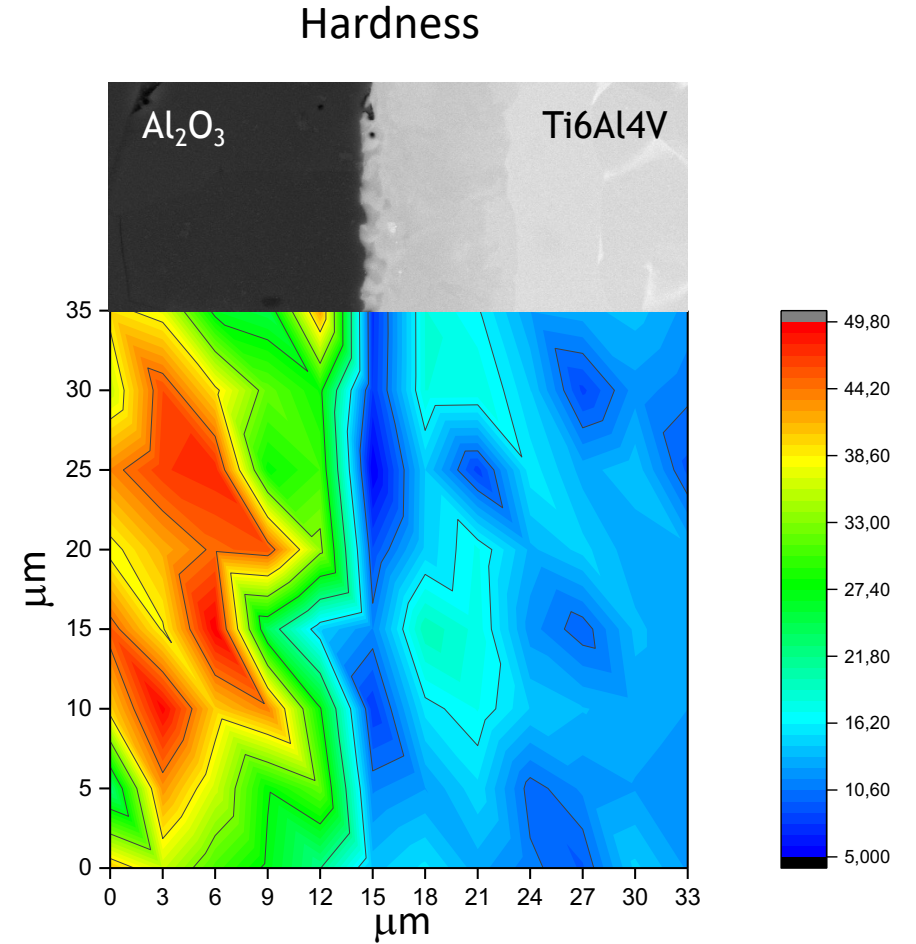
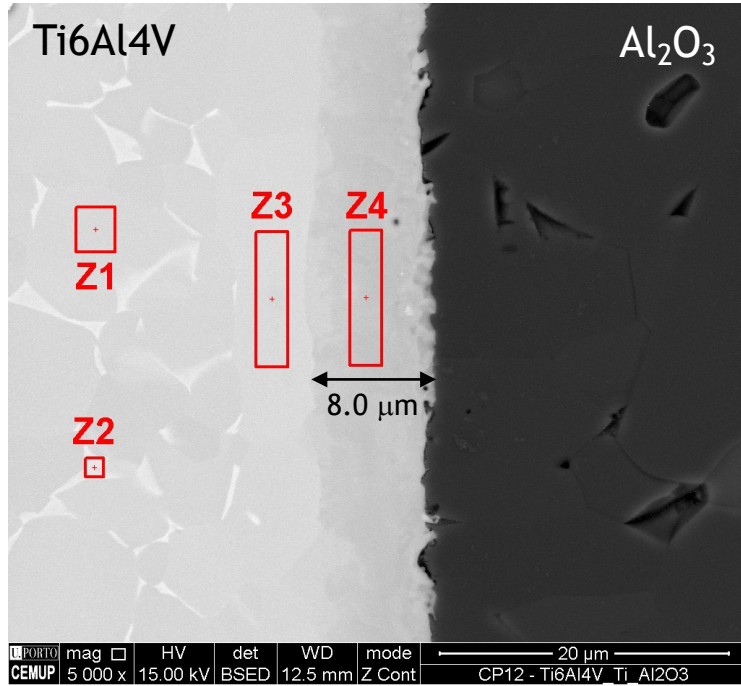
Matrix 8x12 L<sub>max</sub> = 5 mN

Distance between columns = 3 μm

Distance between rows = 5 μm

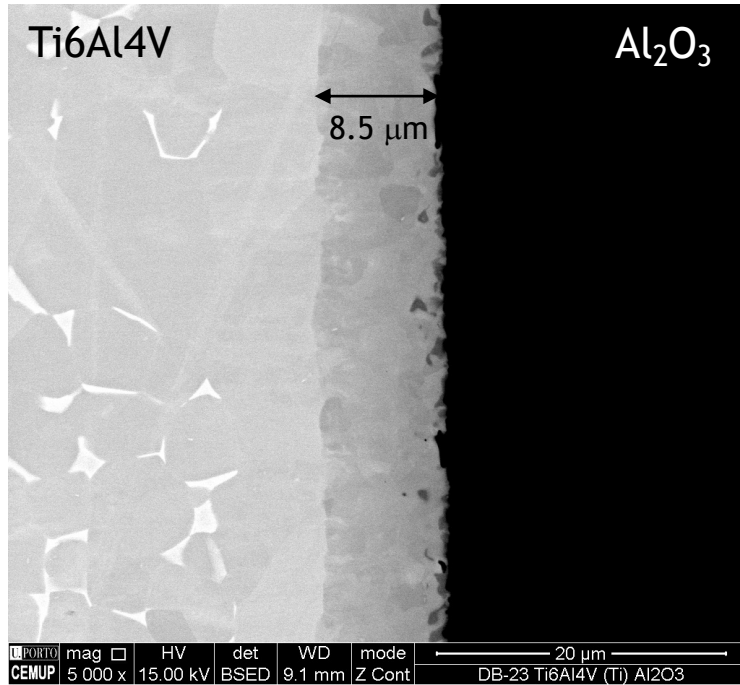


Ti thin film (1 μm) @ T = 1000°C (Contact/ 10 min)

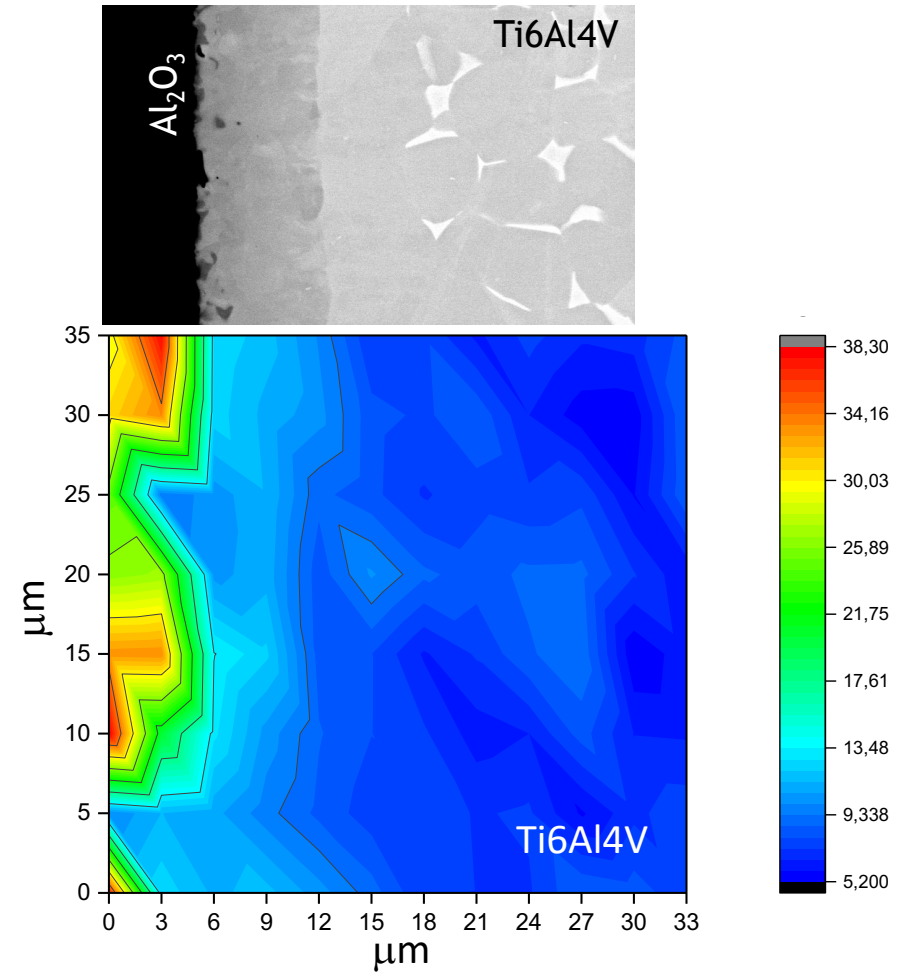


	Ti (at. %)	Al (at. %)	V (at. %)	Possible Phases
Z1	86.8	11.6	1.8	α-Ti
Z2	73.3	7.0	19.3	β-Ti
Z3	86.0	11.9	2.1	αTi
Z4	75.0	25.0	—	Ti <sub>3</sub> Al

Ti thin film (1 μm) @ T = 950°C (Contact/ 60 min)

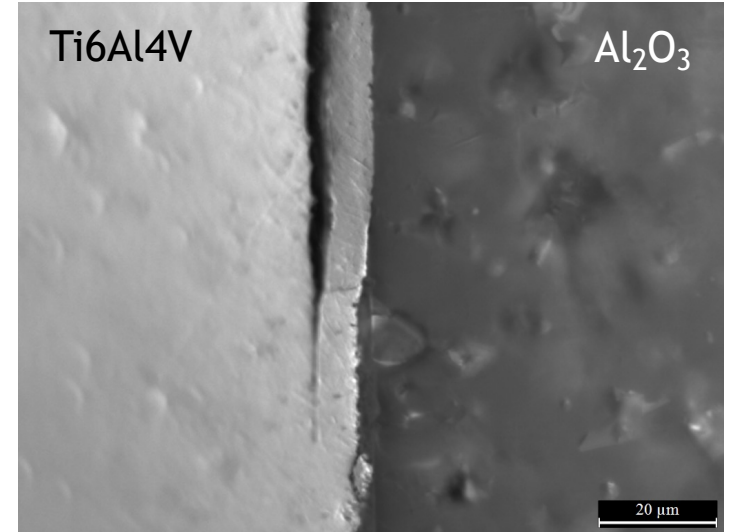
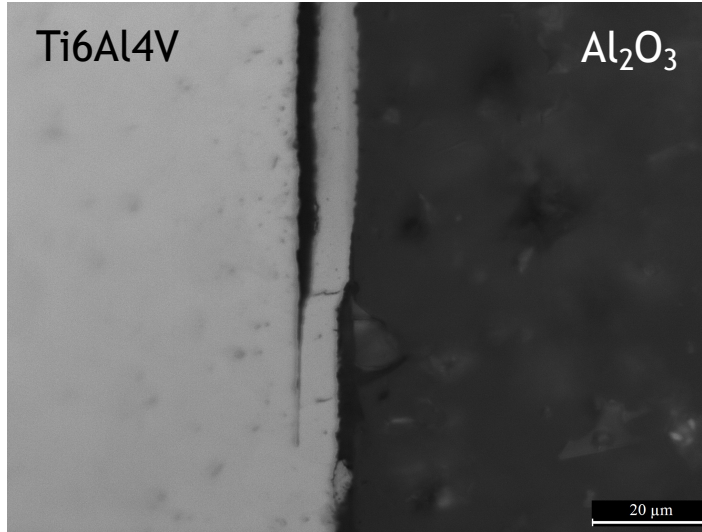
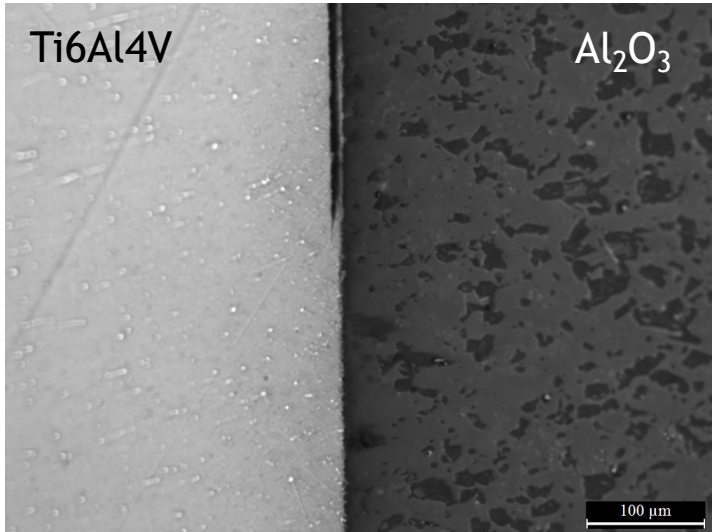


Hardness



Ti thin foil (5 μm) @ T = 950°C (Contact/ 60 min)

### Optical Microscopy



Using the same diffusion bonding parameters, joining was promoted using a 1 μm thick Ti sputtered film deposited onto alumina

Different strategies have been successfully used to diffusion bond Ti6Al4V to Al<sub>2</sub>O<sub>3</sub>

- Reaction-Assisted Diffusion Bonding using Ni/Ti multilayer thin films with nanometric period allowed sound joints to be obtained at 800 and 750°C, by applying 5 MPa during 1h.
- Due to the diffusion of Ni from the multilayers towards Ti6Al4V, the zone adjacent to this base material is enriched in Ti, promoting the formation of NiTi<sub>2</sub>.
- The influence of the multilayer period ( $\Lambda$ ) is not significant, although decreasing  $\Lambda$  results in more homogeneous interfaces.
- Using monolithic thin films as interlayer material sound joints were obtained at 1000 and 950°C, just by putting the Ti coated alumina in contact with the Ti6Al4V base material.
- Due to diffusion a reaction zone constituted by titanium aluminides forms, whose thickness increases with temperature and holding time.
- The hardness and reduced Young modulus maps obtained by nanoindentation corroborate the microstructural characterization and allowed the different reaction layers composing the interface to be clearly distinguished.

This work was financially supported by Project NanoTiC (POCI-01-0145-FEDER-031579) funded by FEDER funds through COMPETE2020 - Programa Operacional Competitividade e Internacionalização (POCI) and by national funds (PIDDAC) through FCT/MCTES.

