Giunzioni metallo-ceramico (YAG-TiAlV): Studio della bagnabilità e della reattività mediante approccio termodinamico

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Giunzione metallo-ceramico (YAG-TiAlV): Studio della bagnabilità e della reattività mediante approccio termodinamico

1. Aim and approach

Optimization of the production of metal-ceramic joints:

- **Wettability study (*sessile drop technique*)**
- **Interfacial reactivity evaluation (*SEM-EDS*, *RX*)**

  Metal drop/ceramic **Ceramic/filler alloy/ceramic or metal**

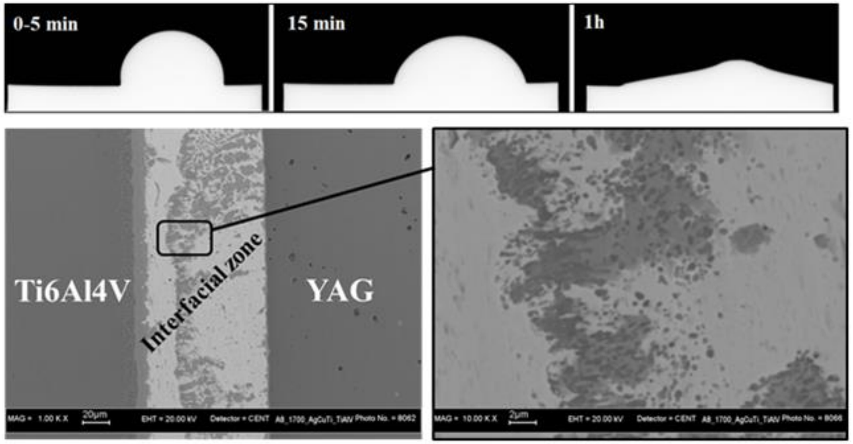
- **Thermodynamic approach (CALPHAD method)**

1. Aim and approach

Experimental work ⟷ **Theoretical work**

- **Wettability**
- **Reactivity tests**

(sessile drop tecnique, SEM-EDS)

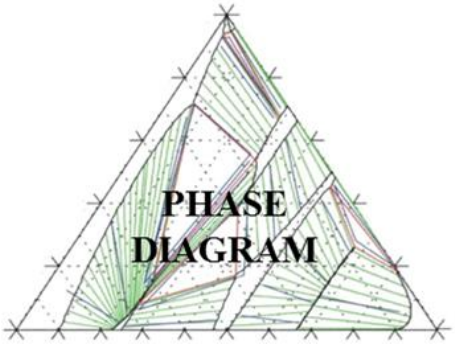


- **Thermodynamic database**

Thermo-Calc

- **Phase equilibria**
- **Element activity**
- **Phase fraction**

- **Choice and redefinition of the filler alloy**

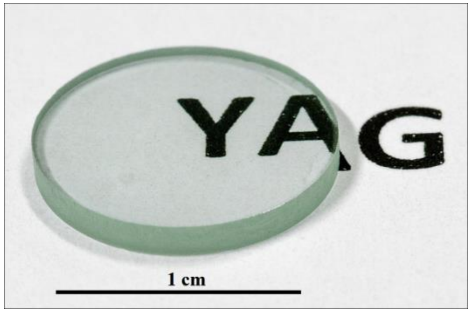


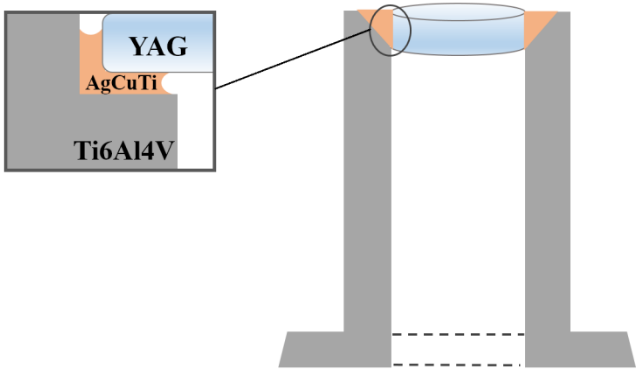
Best joint performances

2. Materials

YAG-Ti6Al4V metal-ceramic joints for marine applications

- **YAG** ($Y_3Al_5O_{12}$) as transparent windows (T~80% vis.) [1]
- **AgCuTi** (AgCu, AgTi, Ag, Cu) as filler
- **Ti6Al4V** as corrosion resistant metallic support



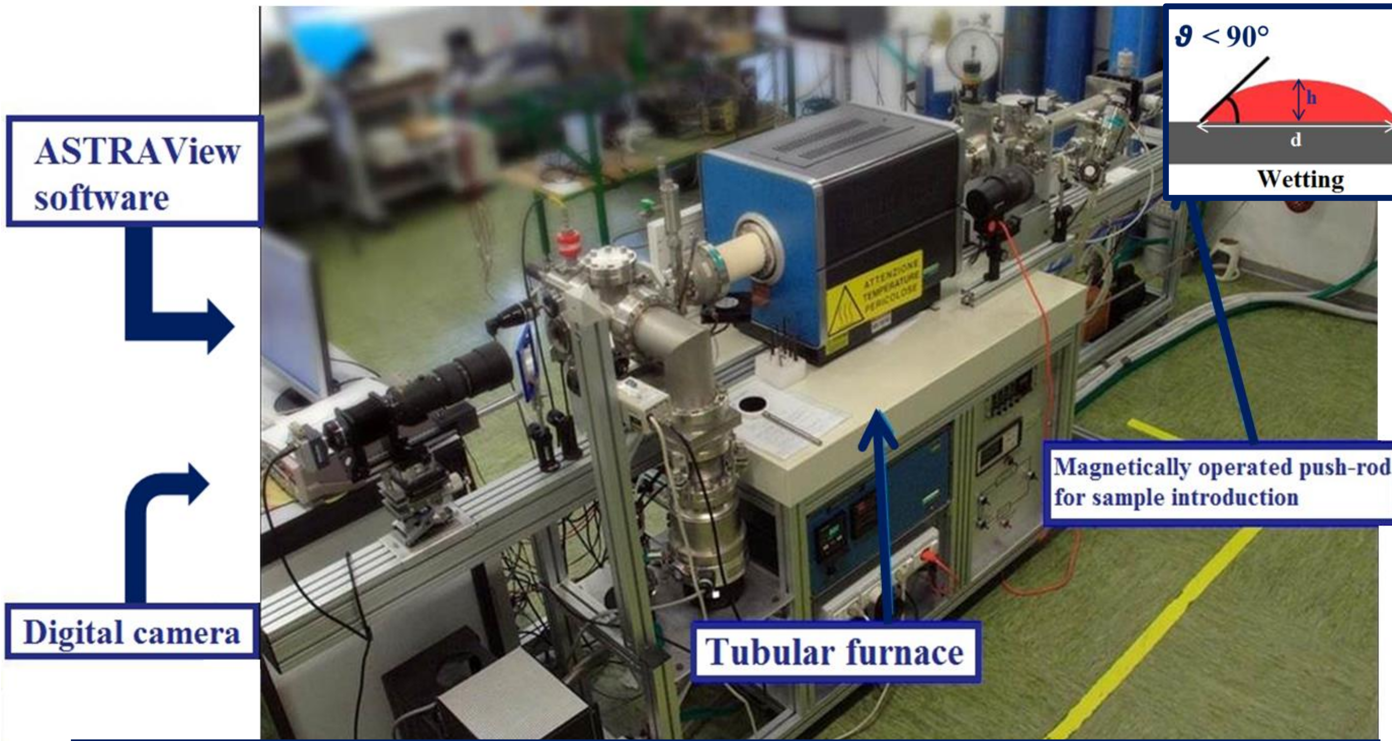


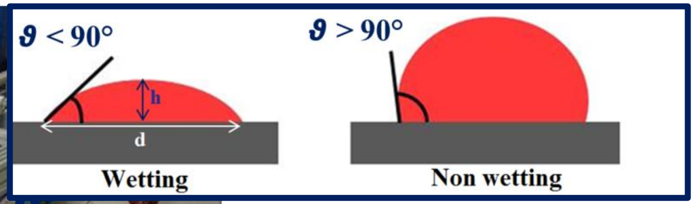
Liquid-based process

- High pressures avoided
- Limited surface preparation
- Complex shapes can be joined

1. J. Hostaša, L. Esposito, D. Alderighi, A. Pirri. Optical Materials. 35, 4 (2013) 798–803.

3. Experimental procedure



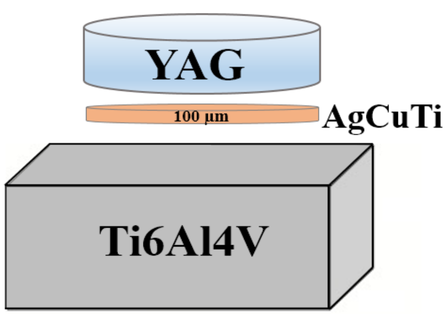


Wetting test:

YAG/filler drop (Ø = 4-5 mm)

Joining test:

YAG / filler / Ti6Al4V



Tubular alumina furnace + optical line + digital camera + SW for measure of contact angles (Tmax: 1550°C)

Experimental conditions:

- Zr getter wrapped around specimens to set PO_2
- Metal/ceramic couple introduced only when all parameters (T, PO_2) = constant
- Fast cooling after wetting test; controlled cooling after joining test (5°C/min)

3. Experimental procedure

Wetting samples

- Holding time: 1h
- Fast cooling

Alloy/metal	T [°C]	ϑeq
Ag	1050	116
Cu	1150	127
AgCu	850	107
AgTi	1050	64
AgCuTi	820, 850, 950	73, 71, 10

Good wettability

↓

Presence of active element

→ *Ti as active element*

Filler alloys compositions

	Ag		Cu		Ti	
	wt%	at%	wt%	at%	wt%	at%
AgCuTi	70.5	57.7	26.5	36.8	3.0	5.5
AgCu	72.0	60.2	28.0	39.8	-	-
AgTi	96.0	91,4	-	-	4.0	8.6

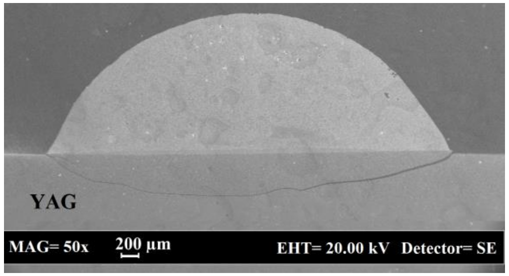
2. Gambaro S., Muolo M.L, Valenza F., G. Cacciamani, L. Esposito, A. Passerone. J. of the Eur. Cer. Soc., 2015

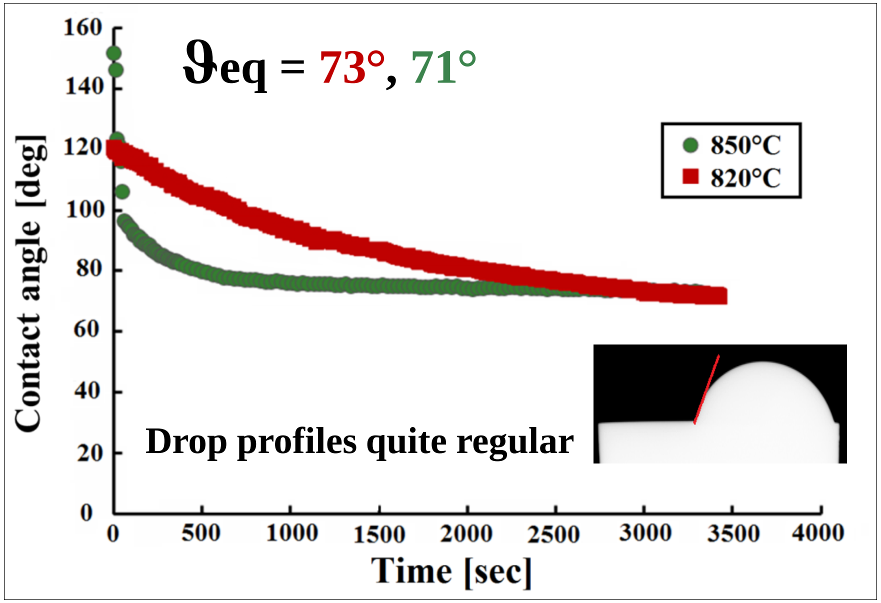
4. Experimental results

AgCuTi/YAG:

T_{test} = 850°C, 820°C

Holding time= 1h
Fast cooling

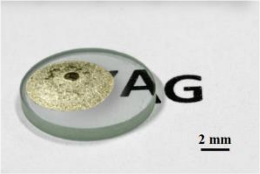


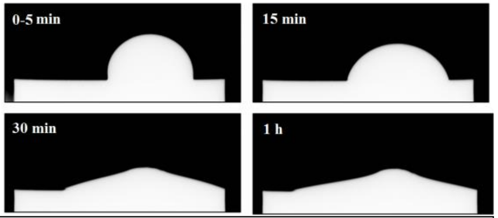


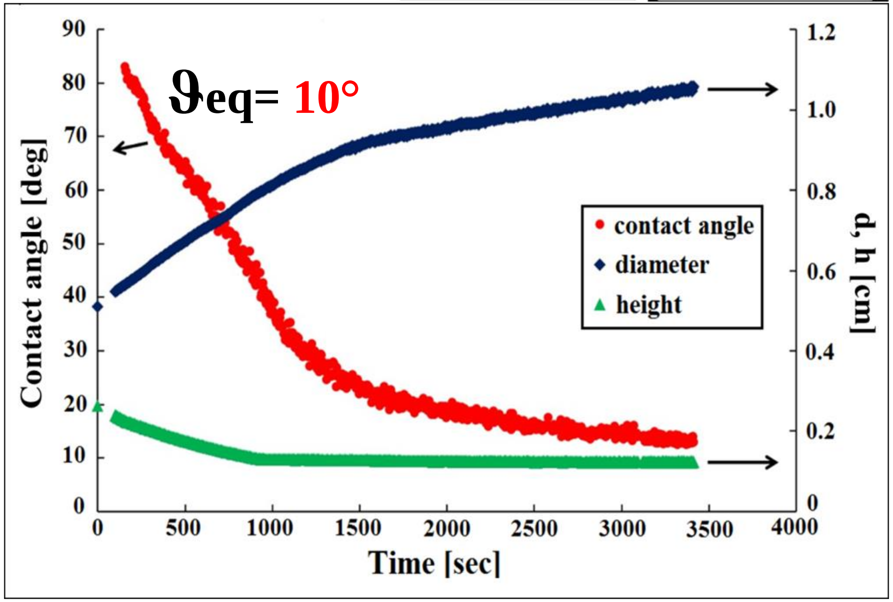
AgCuTi/YAG:

T_{test} = 950°C

Holding time= 1h
Fast cooling

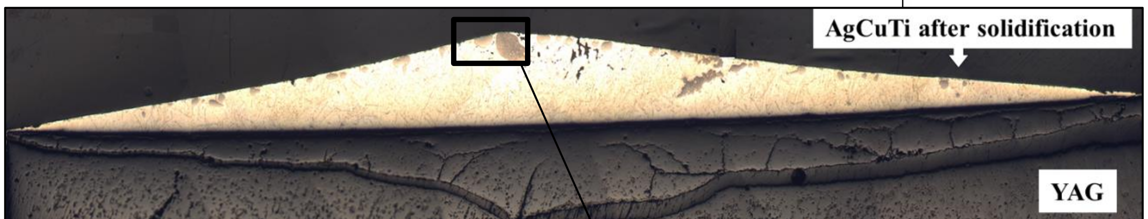


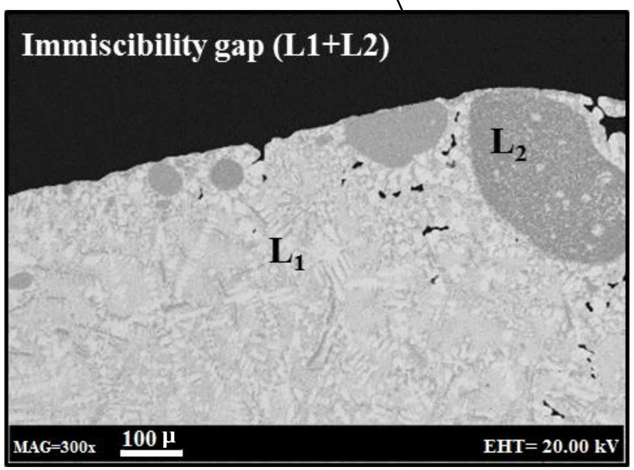


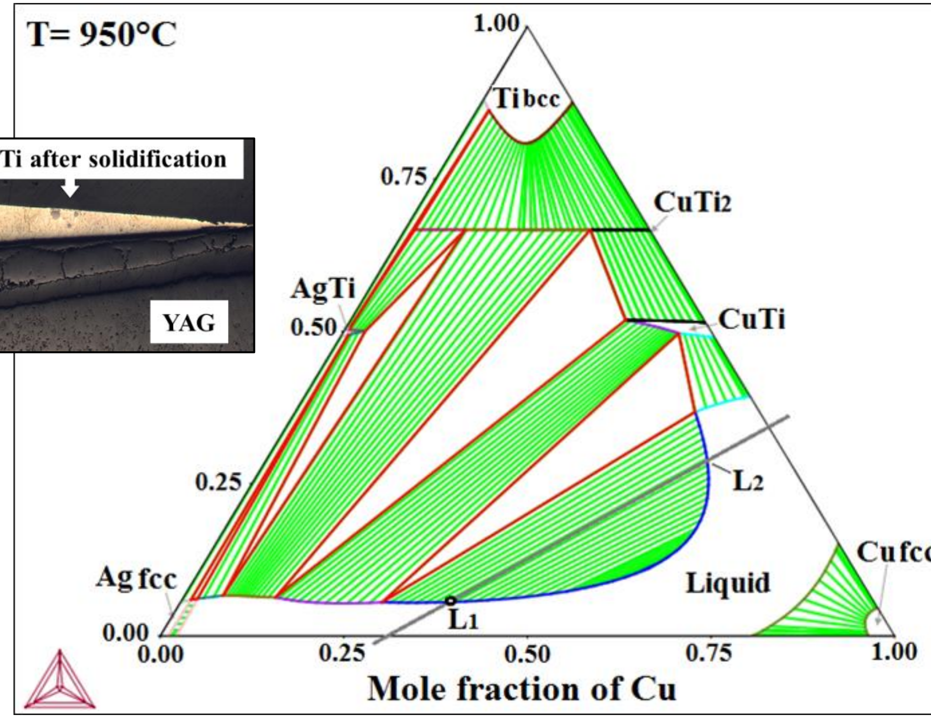


4. Experimental results

AgCuTi/YAG wetting samples at 950°C



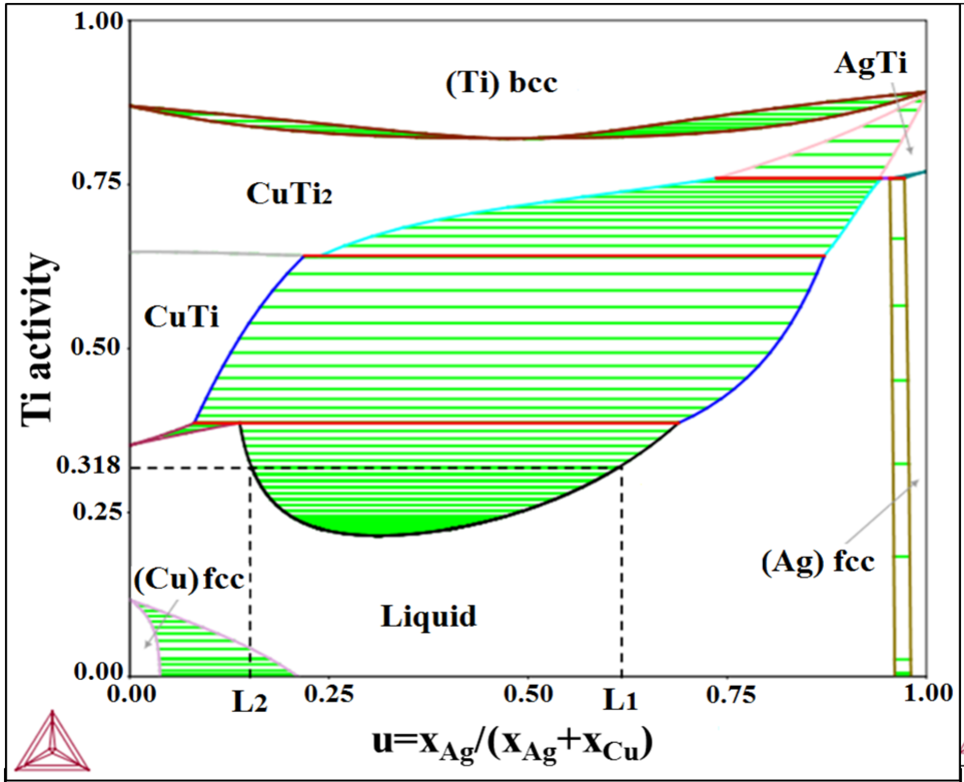


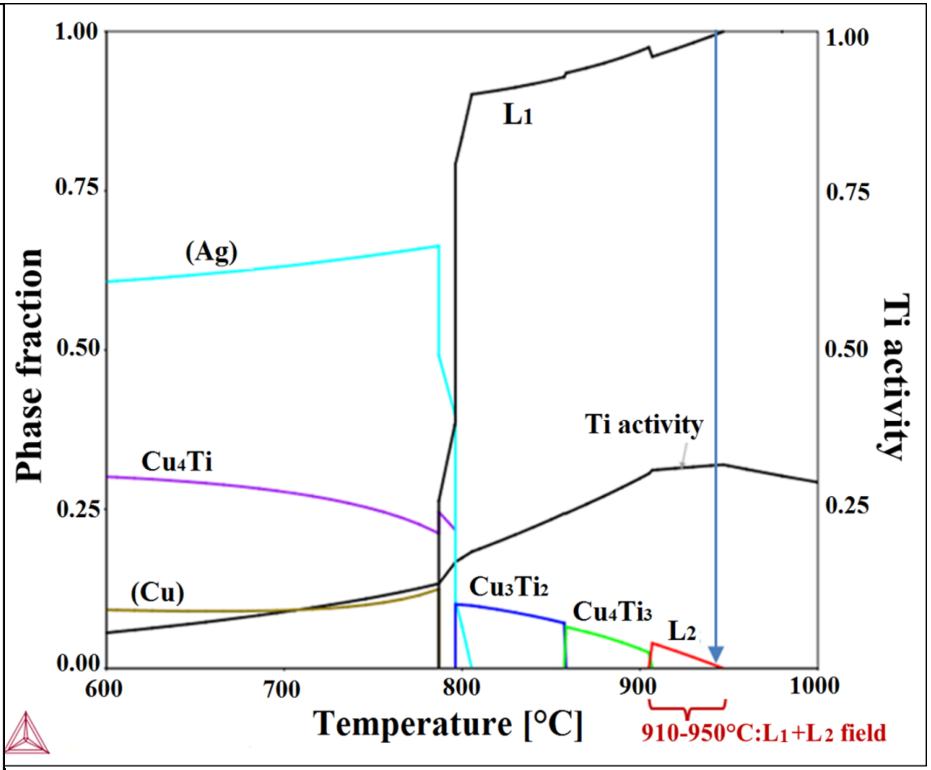


Comp.at%	Ag	Cu	Ti	ϱ[g/cm^3]	a_{Ti}	Υ_{Ti}
Starting alloy	57.7	36.8	5.50	9.9		
L^*_1	57.24	36.72	5.58	8.6	0.318	5.70
L^*_2	11.36	60.45	28.18	6.6	0.318	1.13

3. Hinryj S., Indacochea J. Chem. Met. Alloys, 2008
4. O. Dezellus et al. International Journal of Research, 2011

4. Experimental results





➢ The interfacial reaction (Ti/YAG) depends on the T and on the Ti activity in the liquid phase

Comp.at%	Ag	Cu	Ti	ϱ[g/cm^3]	a_{Ti}	Υ_{Ti}
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3. Hinryj S., Indacochea J. Chem. Met. Alloys, 2008
4. O. Dezellus et al. International Journal of Research, 2011

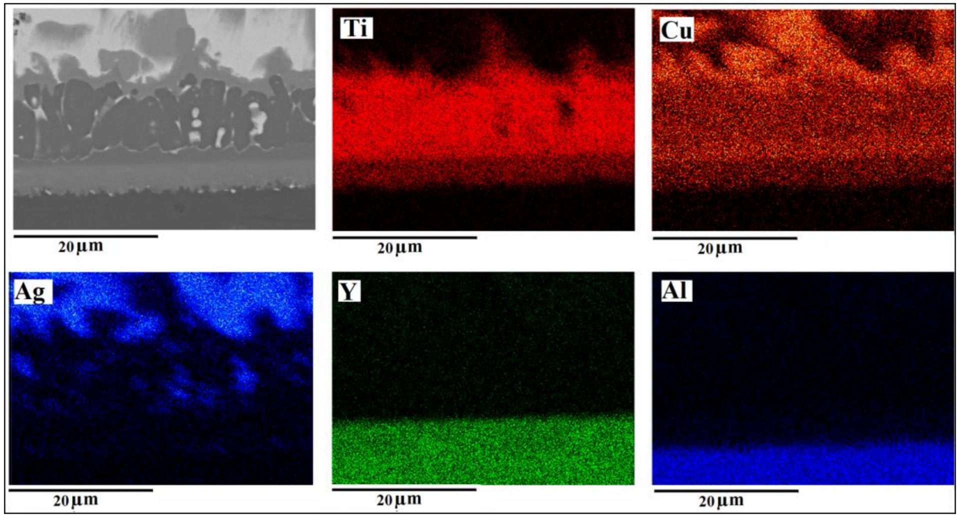
4. Experimental results

AgCuTi/YAG (950°C)

Layer	Composition (at%)						
	Ag	Al	Cu	O	Ti	Y	
YAG	-	26	-	64	-	15	
1st	**0.6**	**3.5**	**16.5**	**41**	**15.2**	**19.8**	
2nd	**1.5**	**2.40**	**35.6**	**18.6**	**41.1**	**0.8**	M_4X [5]
3rd	2.5	0.8	33	-	63.7	-	$CuTi_2$
4th	2.3	0.8	72.5	-	24.4	-	Cu_4Ti

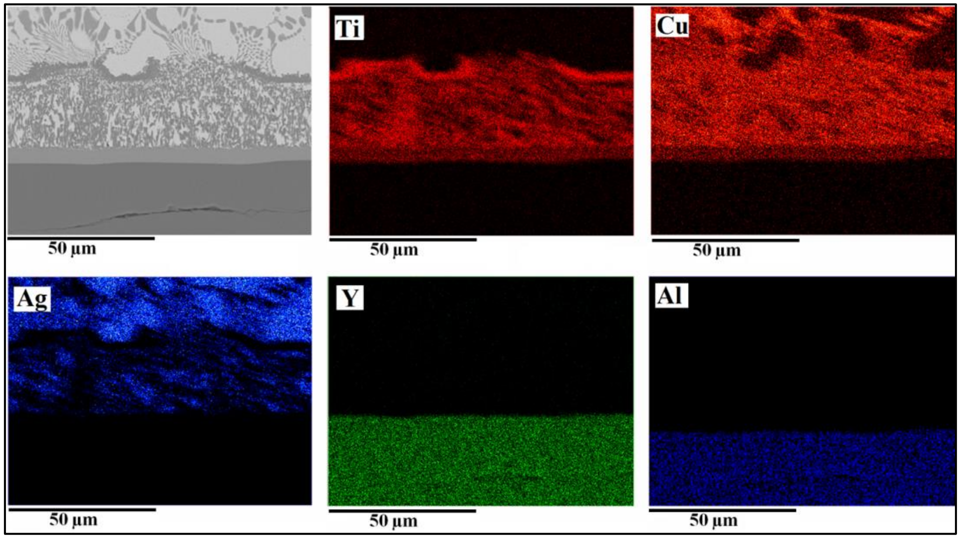
M_4X ($M_{23}X_6$) metal-rich ceramic compound associated to the significant wettability improvement

5. A.H. Carim. Scripta Metallurgica et Materialia 25 (1991)



AgCuTi/YAG (850°C)

Layer	Composition (at%)						
	Ag	Al	Cu	O	Ti	Y	
YAG		25.3	0.3	58		16.4	
1st	**0.4**	**3.1**	**15.7**	**44**	**17.2**	**19.6**	
2nd	**83.6**		**12.9**		**3.5**		**(Ag)**
2nd	3	2.3	50.5		44.2		CuTi
3rd	2.5	2.7	45		49.8		CuTi
4th	60		39.1		0.9		Ag-Cu



4. Experimental results

Joining tests:

Samples	T [°C]
YAG/AgCuTi/Ti6Al4V	850
YAG/Ag/Ti6Al4V	1000

A. Brazing with active filler alloys:

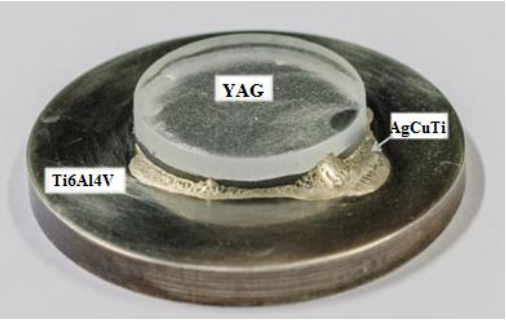
AgCuTi: Ti as active element

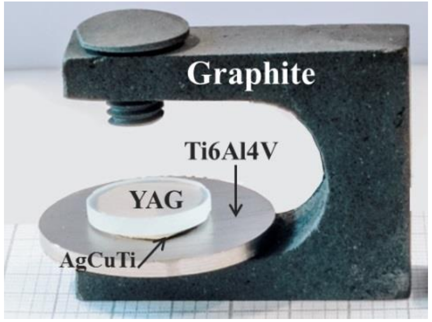
- **Higher process cost**
- **Limited Ti diffusion**

B. Alternative joining approach:

Active-metal-free filler alloys: pure Ag

- **Ti diffusion from the metallic support**
- **'Infinite' source of Ti**



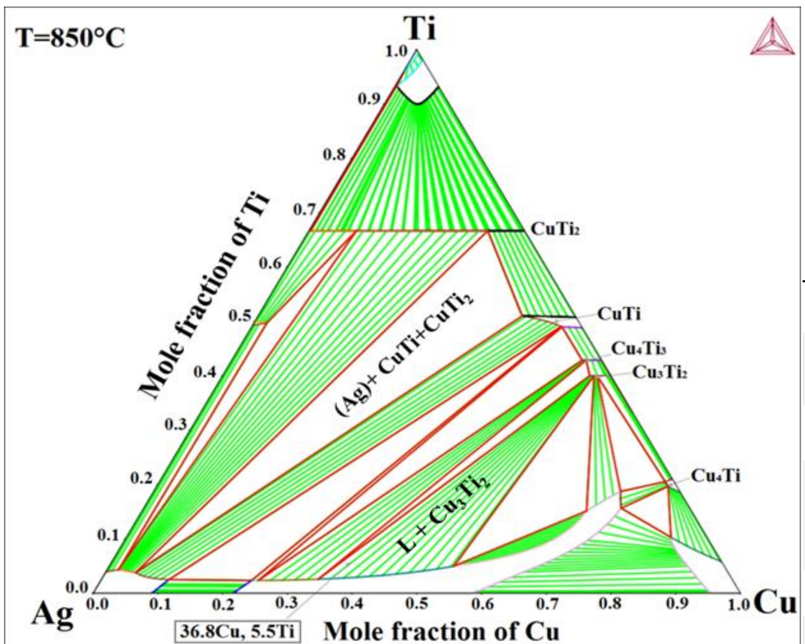


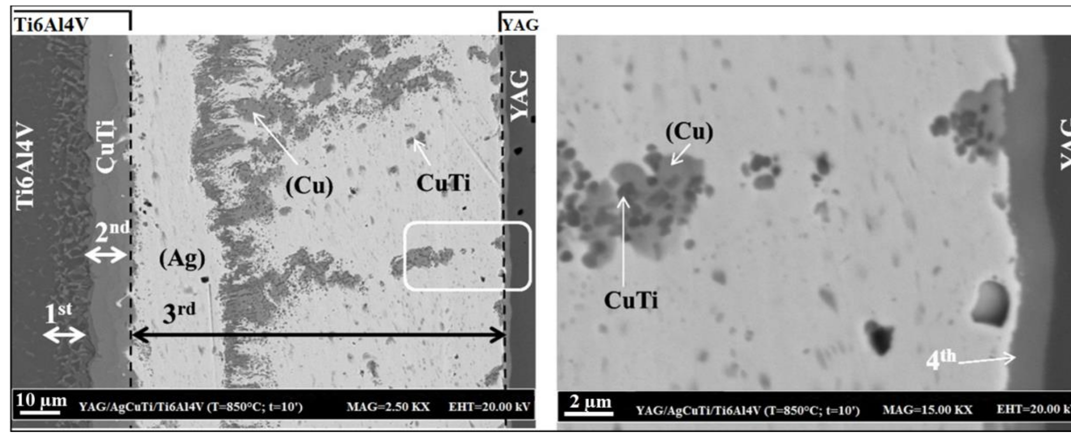
6. Gambaro S., Valenza S., G. Cacciamani, Muolo M.L., A. Passerone. "Brazing transparent YAG to Ti6Al4V: reactivity and characterization" Submitted., 2016

4. Experimental results

YAG/AgCuTi/Ti6Al4V (850°C)

- No fractures or defects along the interface
- Continuous interface
- Well distributed microstructure

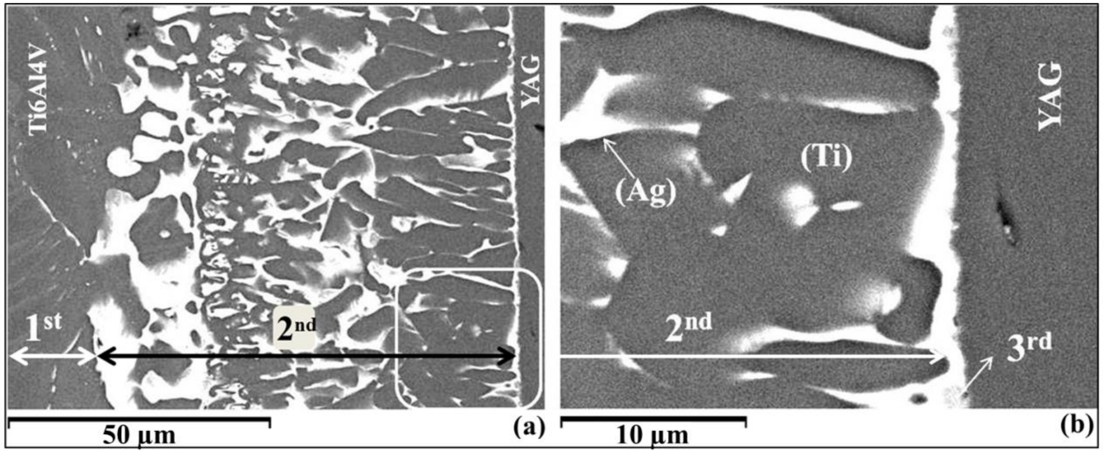


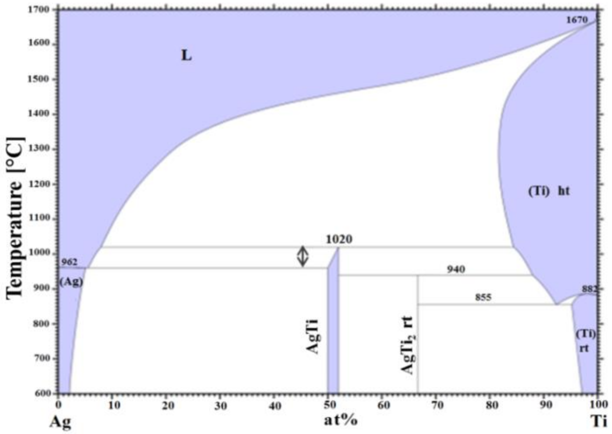


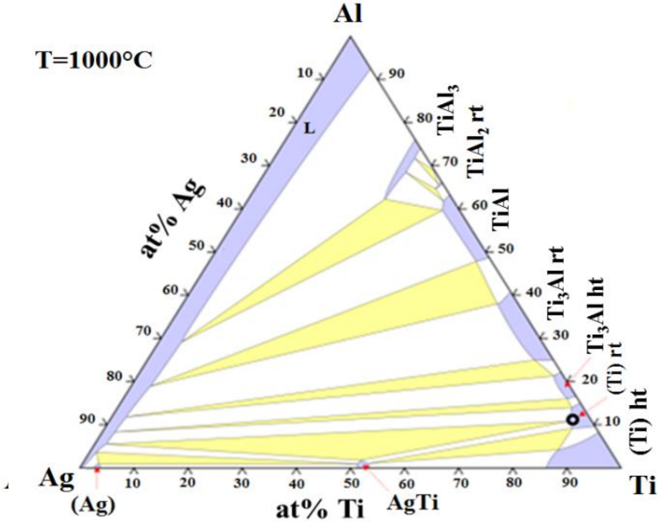
Layer		Thickness	Ag	Al	Cu	O	Ti	V	Y
Ti6Al4V substrate			-	10.5	-	-	86.0	3.5	-
1st	light phase	10 µm	2.0	10.0	20.0	-	66.0	2.0	-
	dark phase		-	20.0	3.0	-	73.0	4.0	-
2nd	CuTi	10 µm	1.5	1.5	49.0	-	47.0	1.0	-
3rd	(Ag)	80 µm	90.0	-	10.0	-	-	-	-
	CuTi		5.3	7.2	42.0	-	45.5	-	-
	(Cu)		3.0	2.0	87.0	-	8.0	-	-
4th		2 µm	1.0	-	24.0	44.0	5.0	-	26.0
YAG			-	25.0	-	65.0	-	-	15.0

4. Experimental results

YAG/Ag/Ti6Al4V (1000°C)







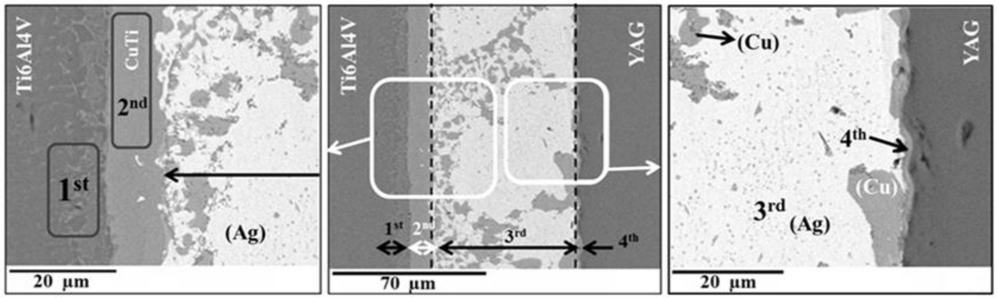
Layer		Thickness	Ag	Al	O	Ti	V	Y
Ti6Al4V			-	10.5	-	86.0	3.5	-
1st		50 µm	2.6	12.4	-	81.0	4.0	-
2nd	(Ag)	75 µm	97.5	-	-	2.5	-	-
	(Ti)		2.0	10.0	-	87.0	1.0	-
3th		2 µm	12.5	3.5	60.5	2.0	-	21.5
YAG			-	25.0	60.0	-	-	15.0

5. Conclusions

Experimental work ⟷ **Theoretical work**

- **Wettability**
- **Reactivity tests**

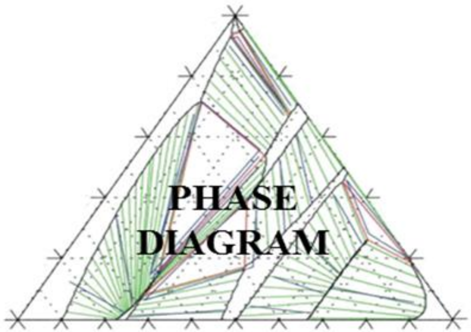
1) Good adhesion
2) Wettability of the ceramic by the filler
3) Wettability of the metallic support by the filler
4) Controlled reactivity



- **Thermodynamic database**
  CALPHAD METHOD

1) Processing temperature
2) Holding time

- **Choice and redefinition of the filler alloy**

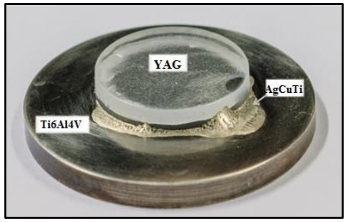


Best joint performances

5. Conclusions

Joining:

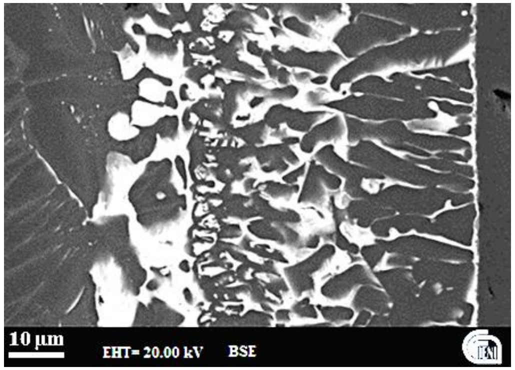
1. **Good joints obtained**



- ➢ YAG/AgCuTi/TiAlV at 850°C
- ➢ YAG/Ag/TiAlV at 1000°C

2. **The Ti amount is not significantly different in the layers in contact with the YAG**

- ➢ About 5 at% with active-filler; about 2 at% with Ag



3. **YAG/Ag/Ti6Al4V**

- ➢ No intermetallic compounds formed at the interface
  Important for the mechanical performance

5. Conclusions

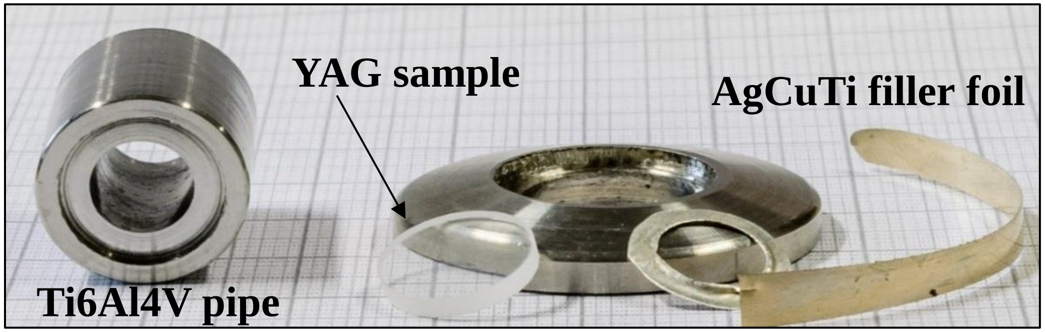
Work in progress:

Deeper evaluation of the joints to determine the best performances:

- **Corrosion resistance tests (marine water)**

IENI-CNR laboratory: Alessandro Benedetti

- **Vacuum tight tests**



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Ritmare
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