

Workshop

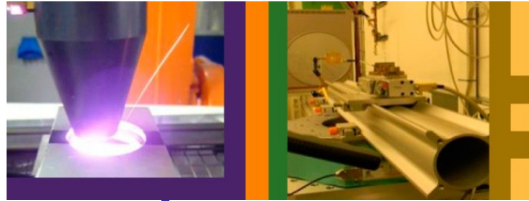
on Processing Characterization
and Applications of Shape
Memory Alloys



Femtosecond Laser Applications in NiTi

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Workshop

on Processing Characterization
and Applications of Shape
Memory Alloys



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Objectives

Analyze beam/material interaction of femtosecond lasers with NiTi based alloys

3 Applications



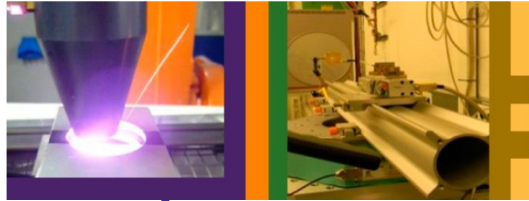
Machining/Cutting



Bonding to glass



Ag nanobrazing to Ti alloy



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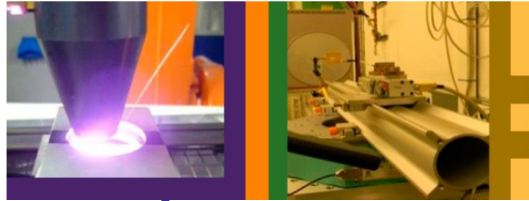
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Introduction

Femtosecond lasers open wide-range new possibilities in microfabrication of metals, polymers, semiconductors, ultrahard materials, transparent materials.

A **femtosecond pulse** (duration below 1ps) in some aspects fundamentally **changes the laser-material interaction** mechanism compared with a long pulse. A femtosecond laser can easily achieve very high peak power, which is powerful enough for full ionization of almost any material.

$1\text{fs} = 10^{-15}\text{s}$ – ultrafast or ultrashort pulse laser



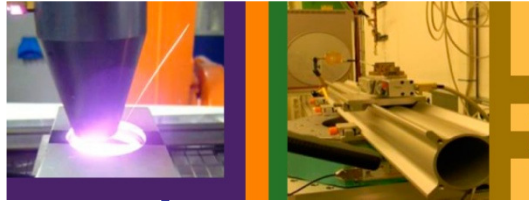
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Introduction

NiTi has been finding increasing applications in industry due to their superior mechanical properties and functional behavior including biocompatibility. As a consequence NiTi is being applied in parts and microparts as in stents, sensors and actuators for biomedics and transport industries.

Microfabrication of these alloys include **material removal, as cutting and drilling, joining, as welding and brazing** where mechanical processes are difficult to use due to the strain hardening properties experienced by NiTi.



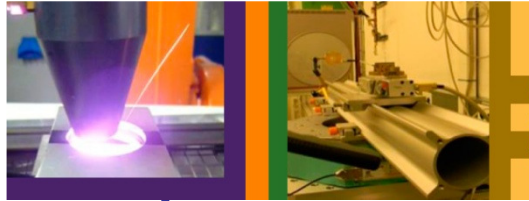
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Cutting NiTi

Femtosecond lasers have been exploited due to its accuracy, precision, but mostly because they **can remove material by an ablative process** in a vapor state with minimal damage of a heat affected zone.

Basically, material decomposition and removal is a consequence of an energy introduced that is above the solid bonding energy. When a very short laser pulse targets a surface a solid ablation can occur in **two stages: absorption** by coupling via multiphoton excitation of electrons in the conduction band and **energy distribution** into the bulk material.



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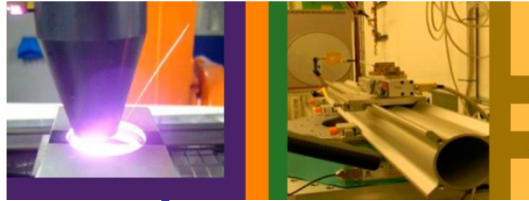
Cutting NiTi

For pulse durations below picosecond, absorption is more dominant than the thermal diffusivity and the heat conduction can be negligible during an ablation process, since evaporation is dominant if the absorbed energy is above the threshold energy for material evaporation (I_{th}). The last one is controlled by the specific heat for evaporation (Q), the material density (ρ), the thermal diffusivity (α) and the pulse duration or interaction time (t) as expressed by eq 1.

$$I_{th} = \rho Q / \alpha t \quad (1)$$

The ablation depth per pulse (L) is, thus, given by Eq. 2 and it increases with the laser intensity for a certain material.

$$L = I / \alpha \ln (I / I_{th}) \quad (2)$$



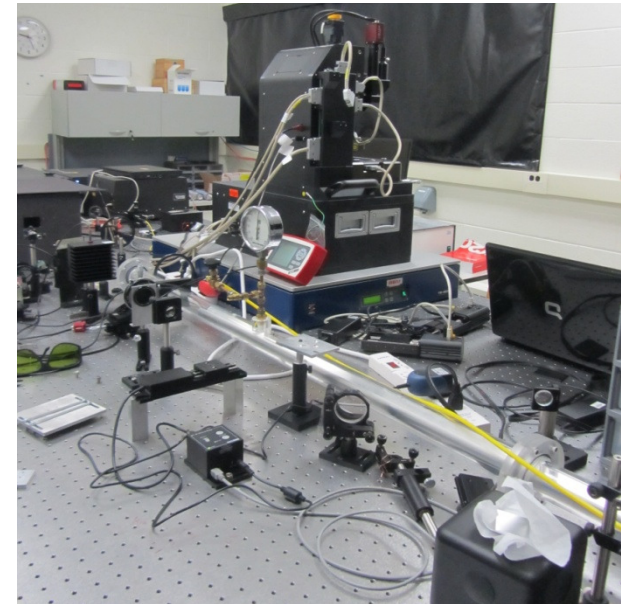
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Cutting NiTi

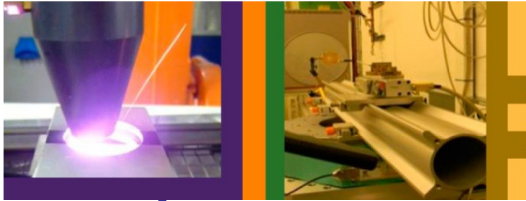
Parameters

1. Polarized laser pulses with 35 fs duration at a repetition rate of 1 kHz
2. Peak power of 2.5 W
3. Wavelength of 800 nm
4. Gaussian beam distribution
5. Travel speed was set at 0.01, 0.1 and 1 mm/s
6. Laser pulses were focused by a lens with 13 cm focal length
7. Mechanical shutter was used to select the number of pulses



Experimental set up. Laser head and fixing system

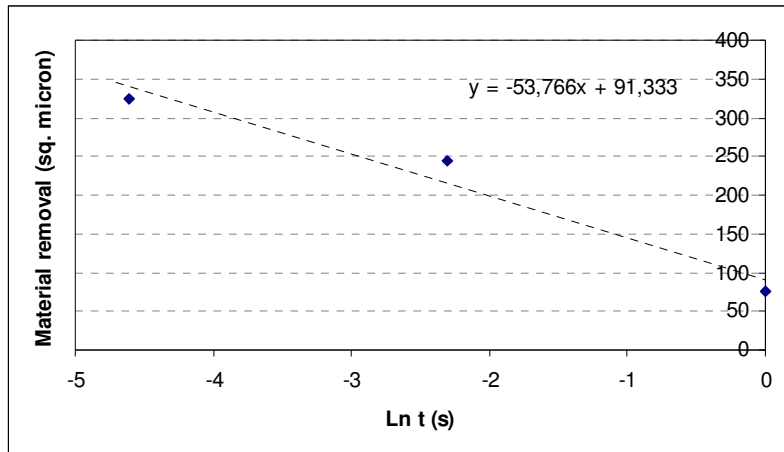
T1 – Sapphire Laser System –
Coherent Inc.



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Cutting NiTi

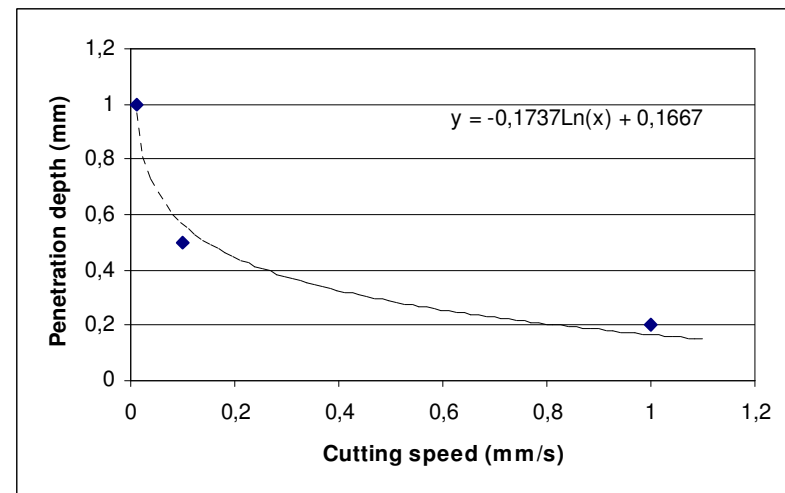


Variation of material removal with the logarithm of the interaction time

$$MR = 91.3 - 53.8 \ln t$$

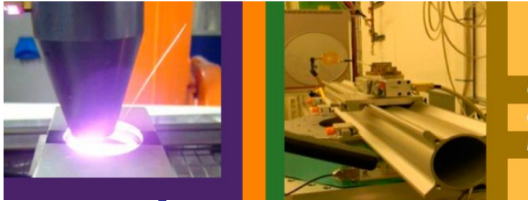
Empirical equation to calculate the material removal rate

$$MR = A + B \ln t$$



$$p = 0.17 - 0.17 \ln v$$

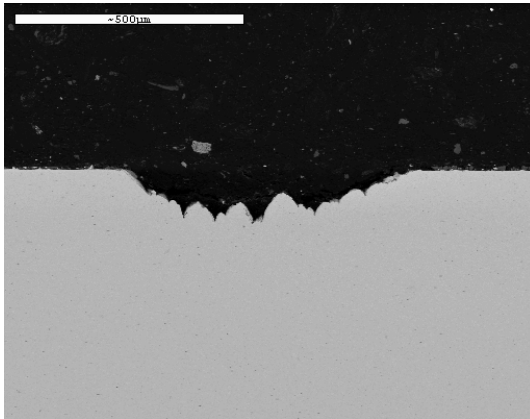
Penetration depth as a function of cutting speed



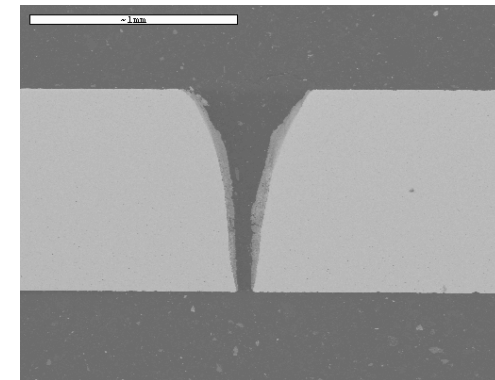
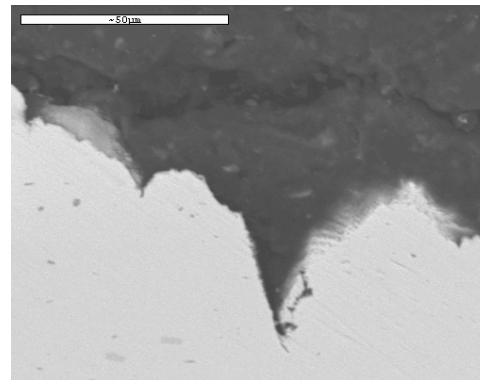
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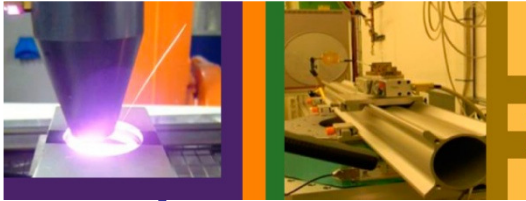


Macrograph of sample machined by femtosecond laser ($v=1$ mm/s)



SEM macrographs of cuts with different travel speeds

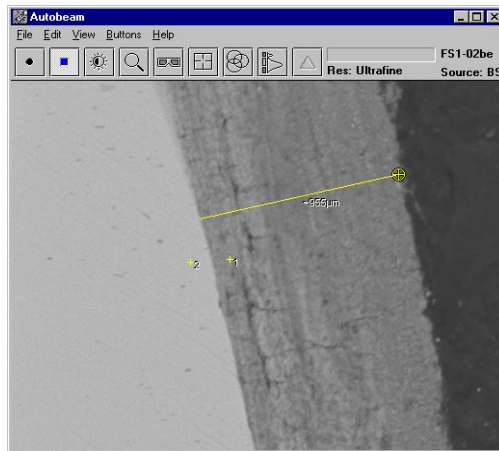
a) $v=1$ mm/s; b) $v=0.01$ mm/s



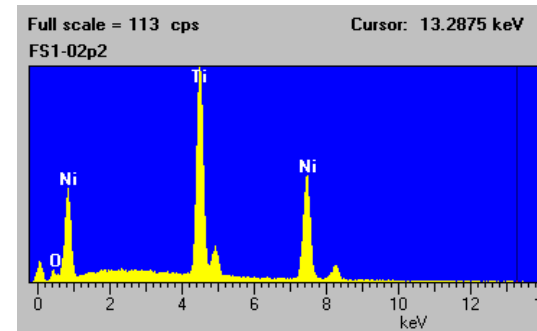
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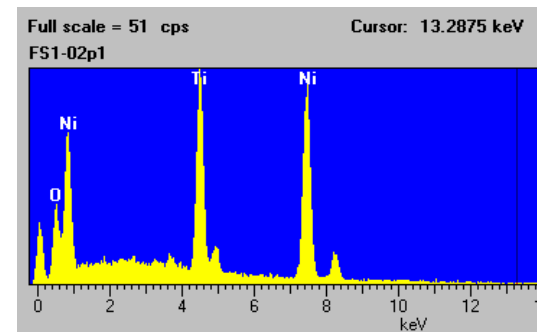
Cutting NiTi



SEM view of a cut surface in NiTi with microanalysis

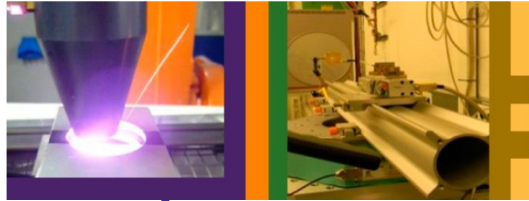


a) EDS spectrum of bulk material



b) EDS spectrum in the resolidified layer

The solid field layer is oxidized and both Ti and Ni are lost in this oxidation process, with a reduction of NiTi.



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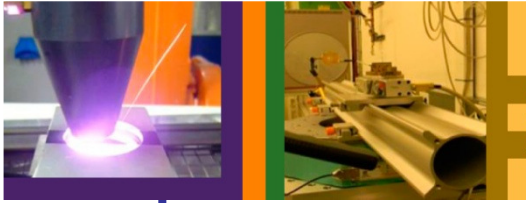
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Cutting NiTi

Conclusions:

From this study it was seen that:

- Femtosecond lasers can cut NiTi with a high precision and a minimal heat affected zone.
- Empirical equations are proposed to calculate the material removal and penetration in ablation of NiTi with femtosecond lasers.
- For low cutting speeds, the surfaces show a resolidified thin layer with solidification cracks and oxidation. This was not observed for high cutting speeds when the interaction time decreased.
- A depletion of Ni was seen which is beneficial specially in biomedical applications, though biocompatibility tests were required to confirm this statement.



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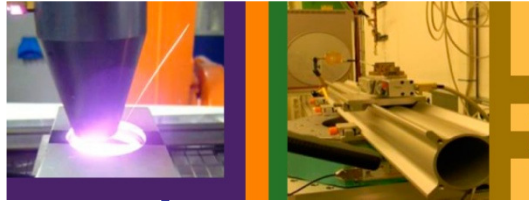
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Bonding NiTi to glass

The scale-down of parts especially for the medical and electronic industry has driven the need to develop new manufacturing technologies, including joining.

The ultra fast pulsed lasers and femtosecond lasers are innovative tools in nanoscale processing.

The aim of the present study is to illustrate and detail the **effects of femtosecond laser irradiation of NiTi onto a glass substrate** identifying the track geometry, the characteristics of the deposited particles both morphological and its chemical composition.



Bonding NiTi to glass

Experimental Procedure

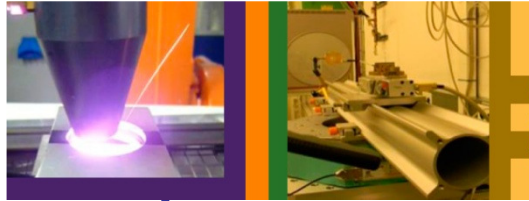
NiTi coupons of 0.4 mm thick were cleaned and etched in a solution of HF:HNO₃:H₂O solution with a dilution of 1:5:10.

An oscillator at 80 MHz, 800nm and 10 fs; an amplifier to 1 kHz, 800 nm, 35 fs, a focal lens holder and a 3D sample stage.

Focal point position and traverse speed were varied. Other processing parameters were kept constant as listed in Table 1.

Laser beam power	1.8 kW
Pulse duration	35 fs
Pulse frequency	1 kHz

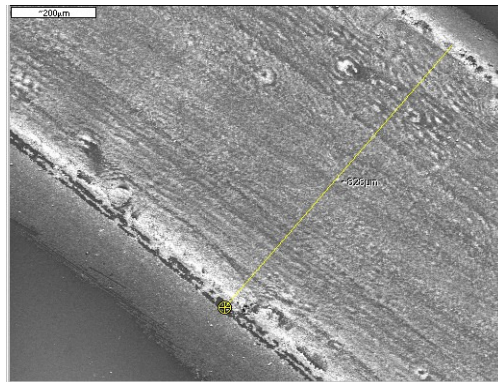
Table 1 – Fixed processing parameters



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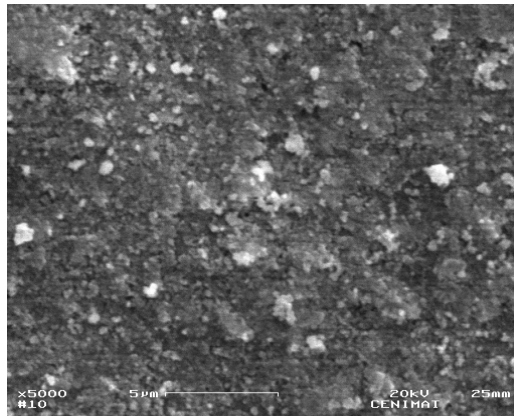
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Bonding NiTi to glass



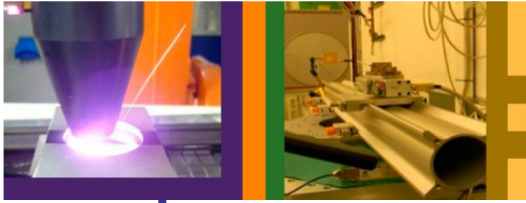
Overall aspect of a laser track

The tracks had about 800 micron width and exhibited a micrometric roughness with a rippling surface structure. The micro strips were seen to be constituted by small re-solidified NiTi droplets that follow a pattern arrangement along the laser traveling direction. The irradiated track borders show a higher particle concentration.



Granular structure in the centre of the NiTi track

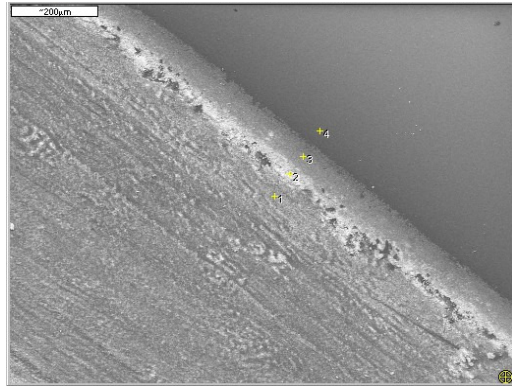
Close to the border of this shockwave impact, the glass substrate was free of particles but no cracking was observed.



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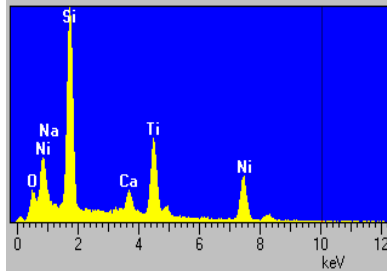
Bonding NiTi to glass



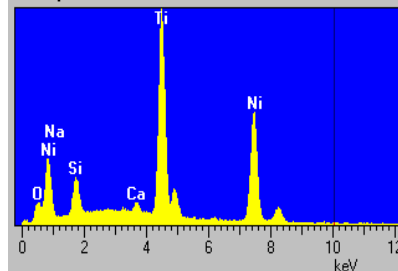
EDS analysis across the track

NiTi in equiatomic form is present along a line starting within the track to the substrate, crossing the boundary area with higher particle concentration.

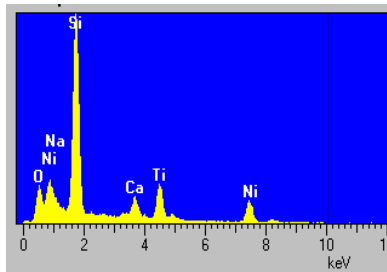
8a



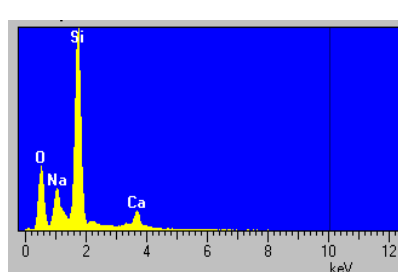
8b



8c

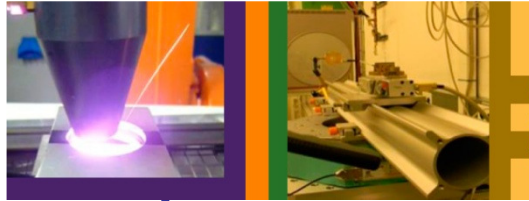


8d



8e

The particle concentration is different in each border suggesting that the beam traveling direction has an effect on the particle distribution within the track.



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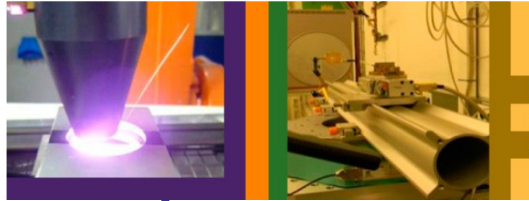
Bonding NiTi to glass

Conclusions:

Glass surfaces can be micro engineered controlling the femtosecond laser process for depositing NiTi particles in a pattern controlled by the laser beam intensity, **proving NiTi can be bonded to glass.**

The laser beam produces micrometric droplets of molten metallic NiTi projected onto the surface by a shockwave produced in two directions, along and perpendicular to the laser traveling direction.

Deposited NiTi particles show a micrometric granular structure. In small areas where this structure does not exist, a dimple surface structure was observed suggesting that a good interface was formed with a ductile rupture mode. After a simple scratch test across a track the deposit was not fully removed suggesting a good adherence to the glass substrate.



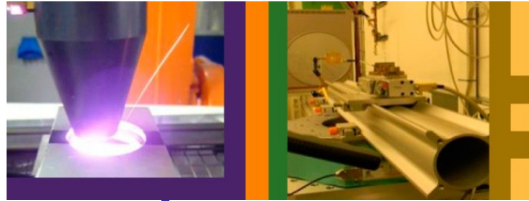
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Ag nanobrazing of NiTi to Ti alloy

Dissimilar joining of shape memory alloys to Ti alloys has long been attempted by several research groups due to the foreseen potential industrial applications. However, the very dissimilar thermo-physical properties of both materials place several difficulties. Brazing can be a solution since the base materials are subjected to a less sharp thermal cycle.

The main goal of the present study was **to assess the potential to use silver nanopaste for brazing** 1 mm thick NiTi to Ti6Al4V plates using different brazing strategies and paste shapes. Analysis of the fracture surfaces and the interfacial microstructure and the characterization of the fracture surfaces was performed.



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Ag nanobrazing of NiTi to Ti alloy

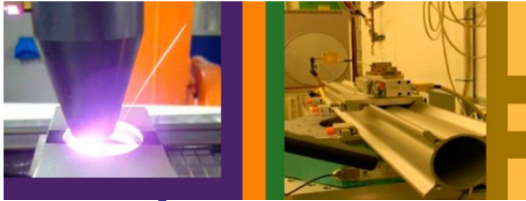
Experimental Procedure

Base materials in this study were equiatomic NiTi and Ti6Al4V alloys. These were cut into strips of 10 mm width, etched in a solution of 7.5% HF + 20% HNO₃ + H₂O and further cleaned with acetone and ethanol to fully remove surface oxides.

Coupons were positioned with an overlap of 3 mm, creating a contact zone of 10x3 mm, in which a nano silver based filler material was applied.

Two types of filler material were used:

- i. Ag paste
- ii. Ag membrane



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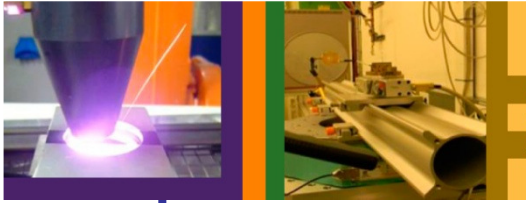
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Ag nanobrazing of NiTi to Ti alloy

Experimental parameters

			Nd-YAG Laser	
	Room temperature 7 days	Compression P=200 N t=60 min	P=1.3 kW F=10 Hz t=3 ms D=3.5 mm v=48 mm/min	P=1.6 kW t=3 ms D= 0.6 mm v=48 mm/min
Silver nanopaste	T1	T2	T4	--
Silver membrane		T3	T5	T6

The results show that achieving a desirable bonding stress is difficult, thus microstructure analysis of the fracture surfaces was performed to find clues to allow for further procedure development.



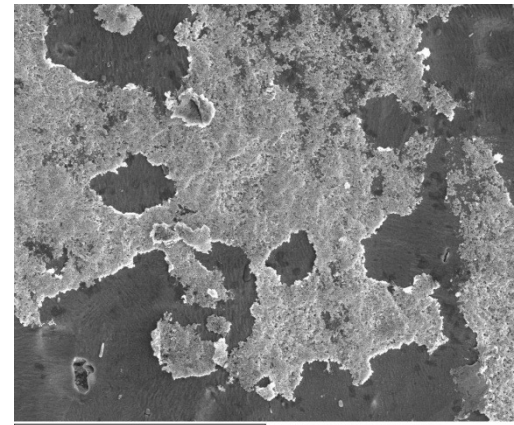
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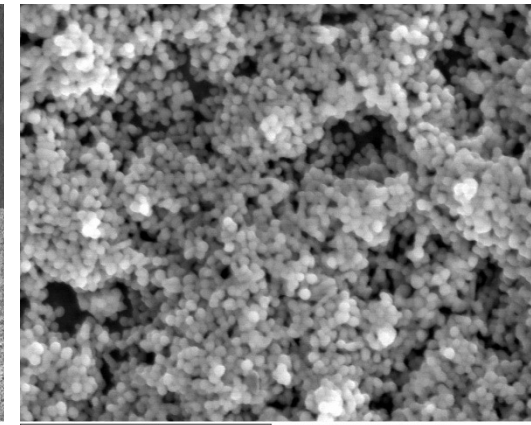
Ag nanobrazing of NiTi to Ti alloy

SEM image of NiTi surface with adherent Ag nanoparticle paste

a) lower magnification; b) higher magnification

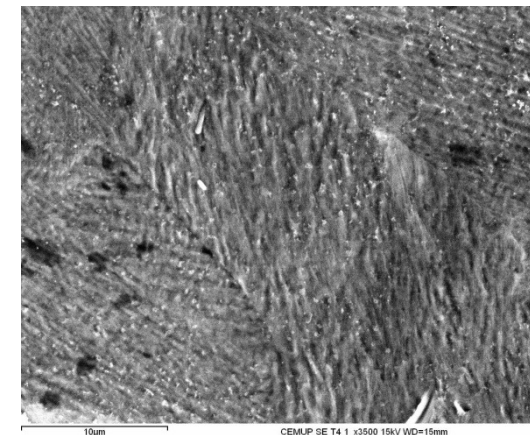


a)

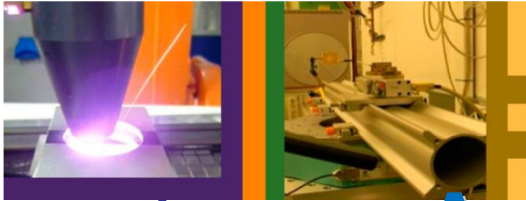


b)

Almost all joints showed adhesive failures or mixed adhesive/cohesive ones in points where adhesion was effective. Figure shows a zone of adhesion of the nanopaste to NiTi. A bright region around the adherent silver is observed and under higher magnification (Fig. b) a dimple structure is displayed which suggests a ductile fracture of the sintered Ag nanoparticles.



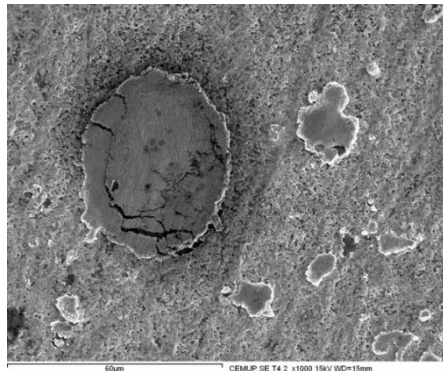
Microstructure of NiTi with a
martensitic phase



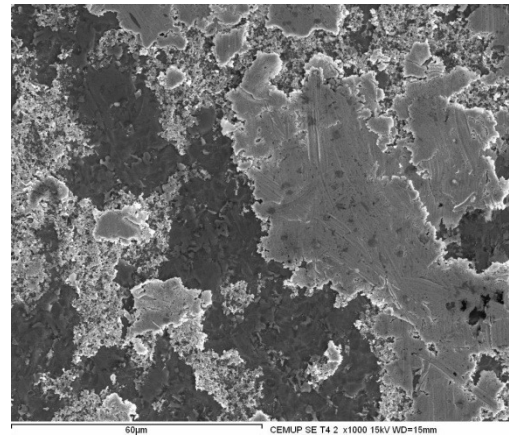
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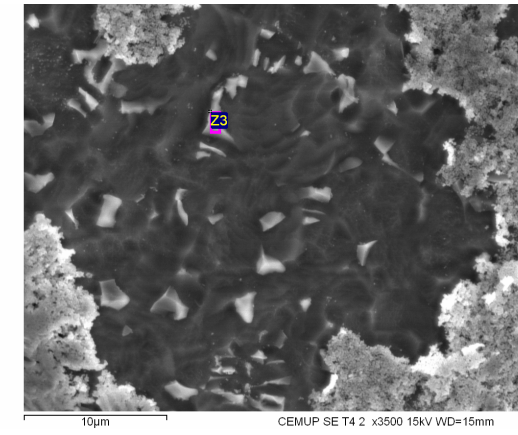
Ag nanobrazing of NiTi to Ti alloy



Melted ag paste
adherent to NiTi

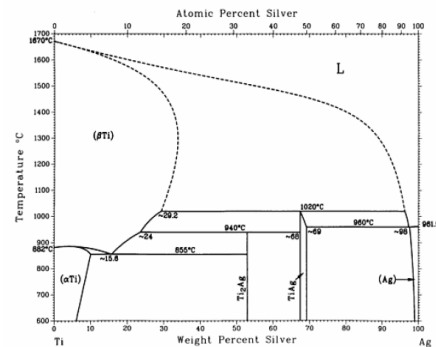


Melted ag adherent to Ti

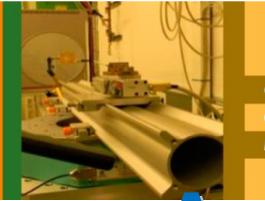


Precipitate in Ti6Al4V

The cracks observed can result from simple solidification cracking of silver with evaporation of constituent water of the Ag nanoparticle paste. This also points to the fact that the paste is more adherent to the Ti alloy than to NiTi eventually due to the existence of the carbide particles.



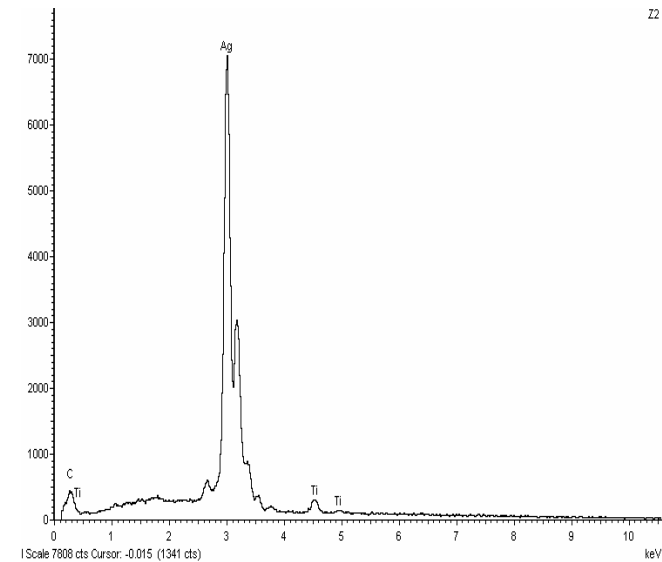
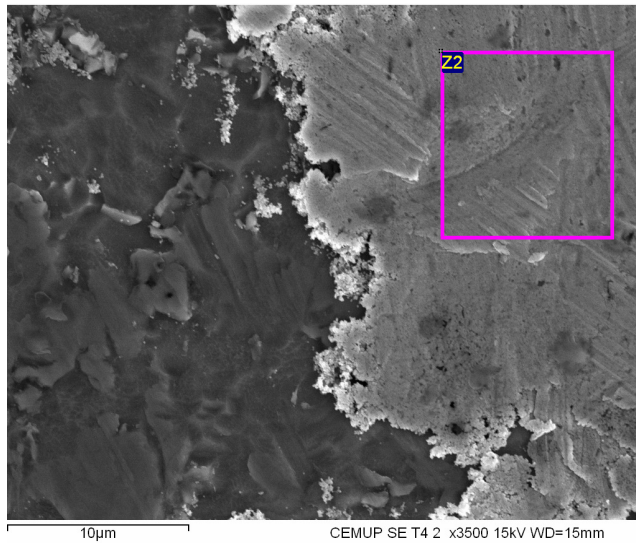
Element	App	Intensity	Weight%	Weight%	Atomic %
	Conc.	Conn.		Sigma	
C K	1.68	0.8024	3.20	0.46	11.38
Na K	0.44	0.8416	0.80	0.15	1.48
Al K	2.75	0.9090	4.62	0.14	7.32
Si K	0.19	0.9751	0.29	0.09	0.45
Ti K	48.22	0.9806	75.19	0.56	67.02
V K	7.58	0.9638	12.02	0.34	10.07
Fe K	1.19	0.9023	2.01	0.24	1.54
Ag L	1.13	0.9336	1.85	0.24	0.73
Totals			100.00		



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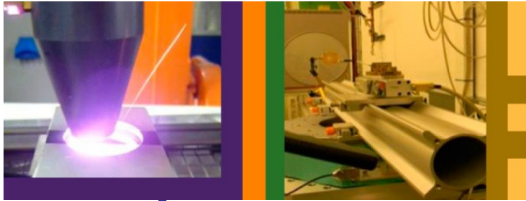
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Ag nanobrazing of NiTi to Ti alloy



EDS analysis of Ag paste adherent to the TiAlV coupon

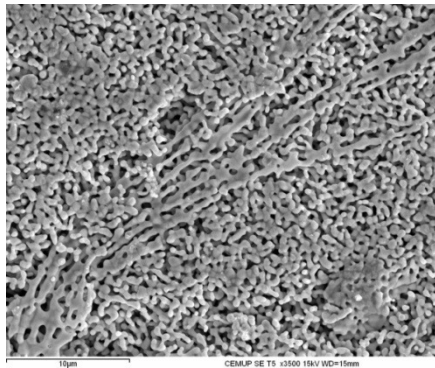
Even in regions where the nanopaste is very thin, there is no migration of Ni into Ti alloy as shown by the EDS spectrum measured on the Ti matrix and the precipitates, that is, the heat generated is insufficient to trigger diffusion of Ni and silver acts as a barrier to atomic mobility.



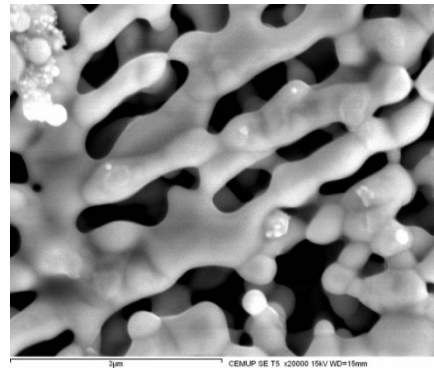
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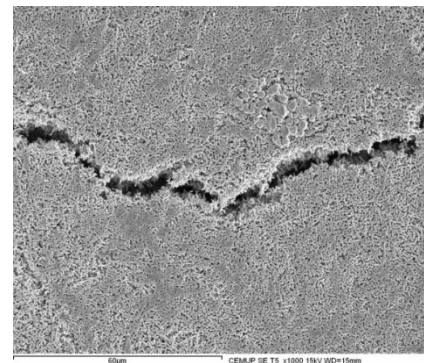


Melted Ag membrane

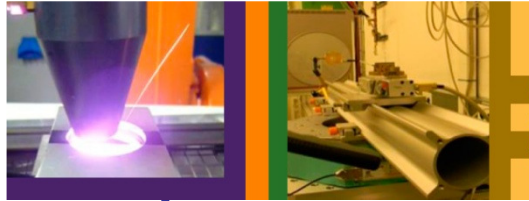


Using the **Ag membrane**, it is observed partial melting of the nanoparticles of the membrane along the laser beam traveling direction. The melted quantity is higher than in the previous trials with the nanopaste and lack of adhesion is observed responsible for the poor joint quality.

Additionally, cracks inside the membrane were also clearly seen where the membrane is seen to crack and is not adherent to none of the substrates.



Aspect of the Ag membrane fracture



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Ag nanobrazing of NiTi to Ti alloy

Conclusions:

Silver nanoparticles were tested for laser brazing of 1 mm thick NiTi to Ti6Al4V

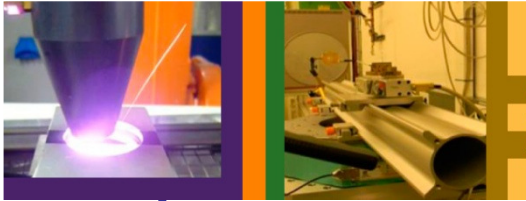
Several strategies for clamping and positioning material plates in lap joint configurations were tested. The overall mechanical strength of the produced joints was low.

No reaction between the silver nanoparticle paste or the membrane with the substrates was triggered in the absence of an external heat source, thus, laser irradiation with different power densities was tested.

From the present work and comparing to published research, it can be concluded that the use of Ag nanopaste as a brazing alloy is limited to small scale parts.

The microstructural analysis of the fracture surfaces revealed insipient melting of the filler, with poor adherence to the substrates. No traces of Ag were seen on the surface substrates suggesting that no surface diffusion occurred to promote joining.

The typical failure surfaces were mostly adhesive, though when using the Ag membrane cohesive fracture mode was also identified under SEM. The fracture types were very similar in all the tests performed.



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Acknowledgments

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Thank you