Experimental Report

Proposal:	**1-04-79**	**Council:**	10/2012
Title:	Eutectoid transformation and silicide precipitation in silicon-doped TiAl-based alloys		

This proposal is a new proposal

Research Area: Materials

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Samples: TiAl+ (Zr,Nb,Mo) + Si (powder)

Instrument	Req. Days	All. Days	From	To
D1B	3	3	15/07/2013	18/07/2013

Abstract:

This project wants to investigate experimentally complex phase transformations at high temperature in TiAl-based alloys. It is a part of a PhD thesis on silicon-doped TiAl-based alloys.

Gamma-TiAl alloys are a subject of growing interest for industrial use after decades of more academic research. General Electric has announced the introduction of Ti-48Al-2Cr-2Nb in its new aeroengine GEnx-1B. But this alloy has limitations in temperature service range, namely due to insufficient creep properties over 800°C. Si-doped TiAl alloys are a potential candidate in aerospace applications due to increased mechanical properties at high temperature.

In this context, the fundamental understanding of the phase transformations starting from single-phased alpha domain over ~1350°C and leading to M_5Si_3 strengthened lamellar or duplex microstructures is crucial. We propose to study five grades : one TiAl+Si ternary alloy, one TiAl+Zr ternary alloy and three quaternary alloys showing the influence of Nb, Mo and Zr alloying both on the eutectoid transformation and the M_5Si_3 formation. For accomplishing this, we apply for 2 days of beam time at D20 running at high flux or for 3 days at D1B.

Abstract

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Experimental methods

The experiments focused on two alloys named Ti-47Al-0.3Si and Ti-47Al-2Zr-0.3Si elaborated by arc melting furnace under vacuum, their chemical composition measured by ICP are given in table 1. For each composition, we have 3 samples corresponding to 3 different microstructures:

- As cast
- Homogenised by a heat treatment at 1400°C for 1h
- M_5Si_3 strengthened, corresponding to the following heat treatment : 1400°C for 1h and 1000°C for 100h

Each sample was bowl milled and sieved at 100 meshes to obtain powder for the diffraction experiments. The ND experiments using D1B instrument were performed using a vacuum furnace and a Nb sample holder. Three different types of experiments are:

- In-situ measurement during the homogenisation of as-cast samples: RT→1400°C→RT
- In-situ measurement during a temperature scan in the M_5Si_3 precipitation range on homogenised samples: 600°C→1200°C
- RT diffraction on homogenised samples and M_5Si_3-strengthened samples.

RT microstructures of as-cast samples consist in lamellar α_2 (DO_{19}) and γ ($L1_0$) grains with the presence of eutectic γ ($L1_0$) - M_5Si_3 ($D8_8$) at grain boundaries. RT microstructures of homogenised samples consist only in lamellar grains. RT microstructures of M_5Si_3-strengthened samples consist in lamellar grains with M_5Si_3 at interlamellar interfaces.

At high temperature (~1400°C), we expected to encounter α (A3) phase, it is to note that due to its disordered character, this phase cannot be detected during ND experiments. Its presence can be deduced from the disparition of the other phases.

Ingot	%at(Ti)	%at(Al)	%at(Si)	%at(Zr)
Ti47Al2Zr0,3Si	52,7±2,6	45,5±2,3	0,29±0,01	2,04±0,1
Ti47Al0,3Si	54,1±2,7	45±2,3	0,31±0,02	0

Table 1

Preliminary Results

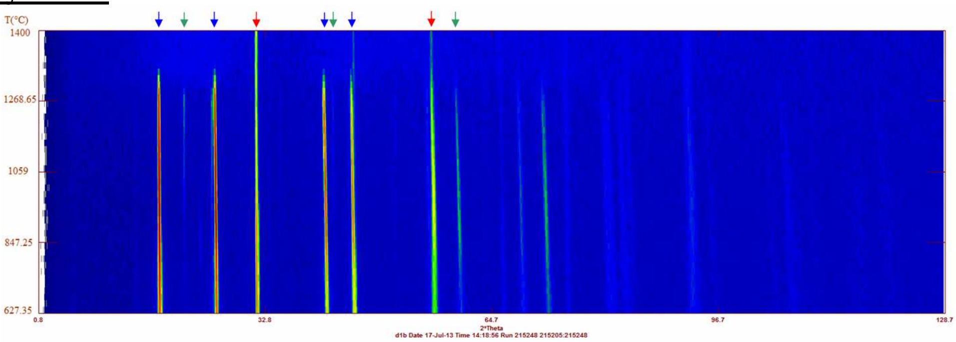

Fig 1: homogenisation on the as-cast Ti-47Al-0.3Si (heating period)

In Fig 1 and Fig 2, we can see the contributions from the sample holder (red arrows), α_2 (green arrows) and γ (blue arrows). We performed Rietveld refinements with Fullprof, the results are presented fig 5. The α single-phased domain is reached at around 1380°C for Ti-47Al-0.3Si and 1360°C for Ti-47Al-2Zr-0.3Si.

The eutectoid $\alpha \rightarrow \alpha_2 + \gamma$ transformation temperature given by the disparition of α_2 seems to be very overestimated. We prefer rely on the γ evolution to estimate this temperature (corresponding to the beginning of the decay of γ contribution), which gives a relevant value of around 1050°C for both samples.

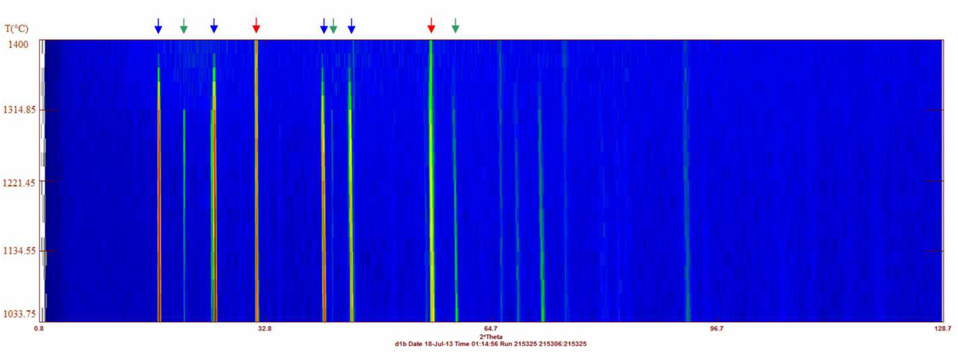

Fig 2: homogenisation on the as-cast Ti-47Al-2Zr-0.3Si

In Fig 3 and Fig 4, we can see a peak of the silicide $D8_8$ phase (white arrow); with Rietveld refinement using Fullprof we can plot the M_5Si_3 contribution. Due to very low intensity, quantitative refinements can't be done for this phase, however, qualitative evolution and temperature range of this phase given by fig 6 is very helpful. The peak of M_5Si_3 volume fraction is observed at 950°C for both alloys. Cell parameters of the phases during this scan are presented Fig 7, 8 and 9.

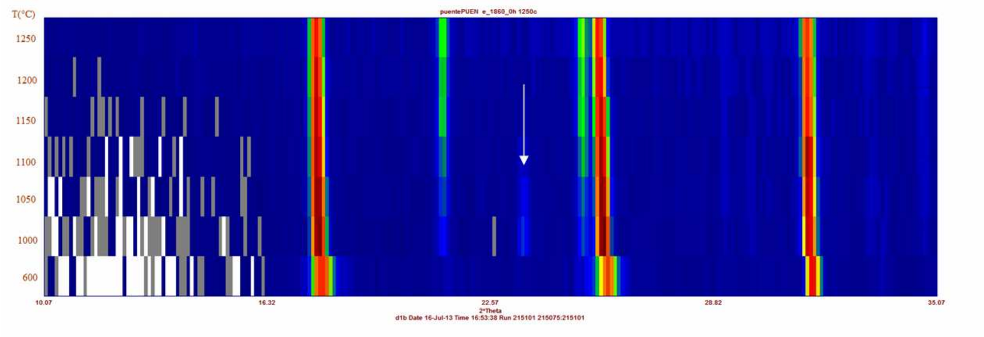

Fig 3: scan on Ti-47Al-0.3Si

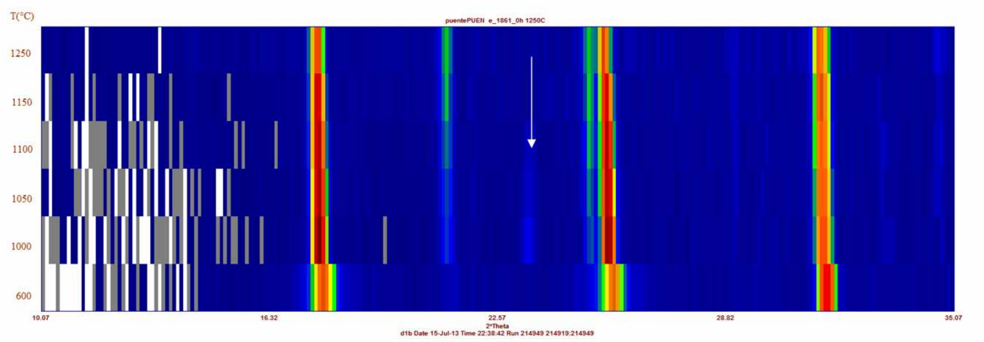

Fig 4: scan on Ti-47Al-2Zr-0.3Si

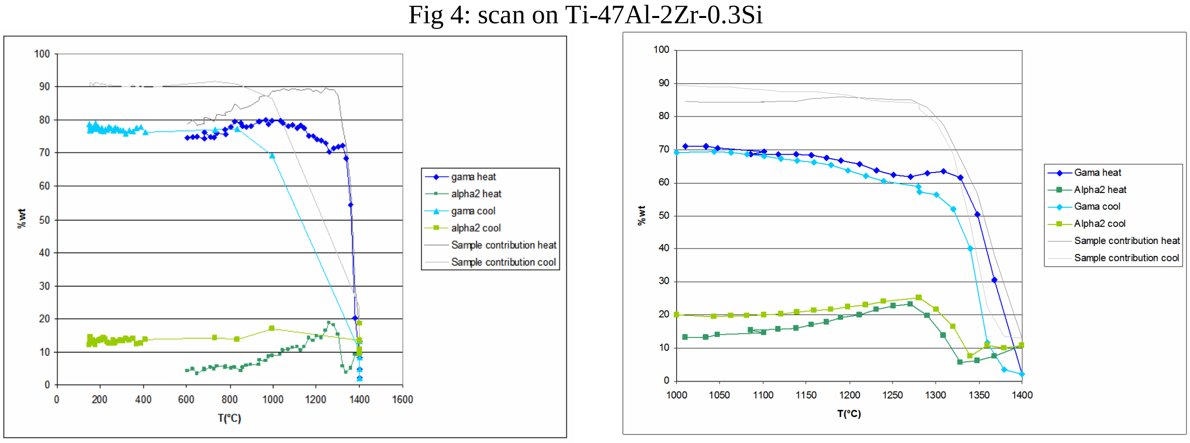

Fig 5: Rietveld refinements on the homogenisation experiments

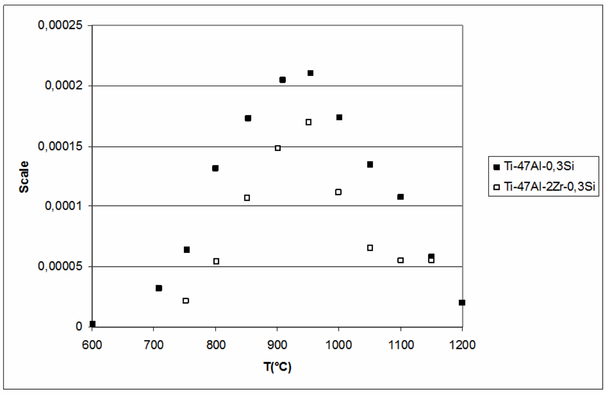

Fig 6: Ti_5Si_3 contribution in the ND

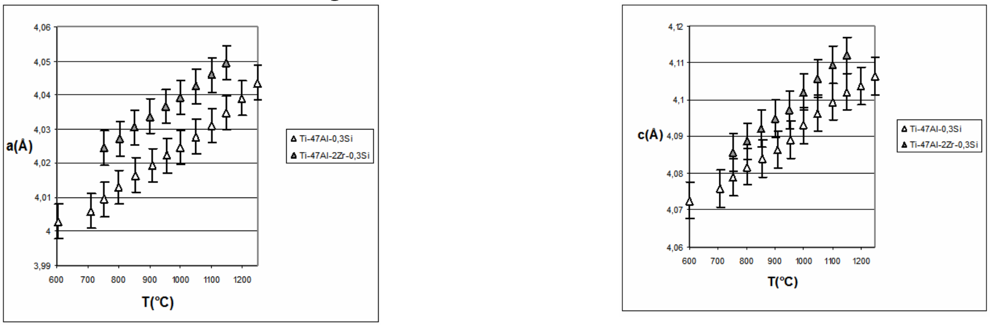

Fig 7: γ cell parameters

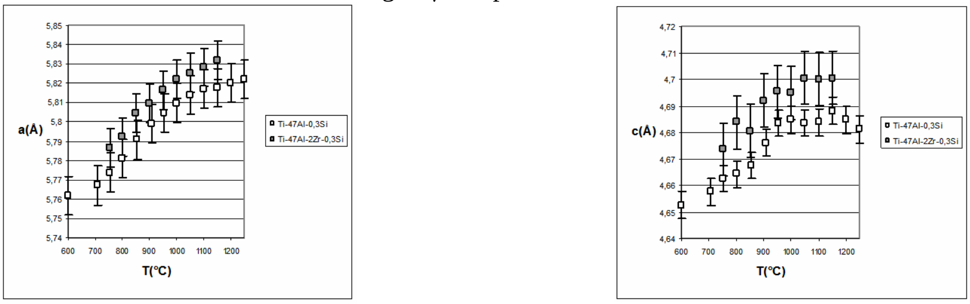

Fig 8: α_2 cell parameters

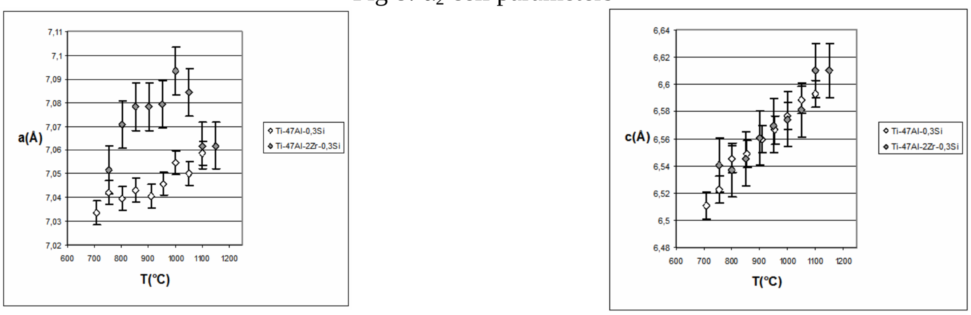

Fig 9: Ti_5Si_3 cell parameters

The RT experiments on M_5Si_3-strengthened and homogenised samples were performed, but due to, we think, an unexpected amorphisation of the samples during the bowl milling, refinements cannot be performed for these experiments. We think a low temperature (~700°C) heat treatment could solve this problem for future diffraction experiments, since, in the case of hot diffraction measurements, the quality of diffraction spectra becomes fairly better at temperatures above 700°C.

This experimental session, even with this last problem concerning only RT experiments, gave us good results on the solid state transformations that took place in our alloys. The observation of the M_5Si_3 precipitation and its dependence to temperature is crucial for microstructural control of silicon-doped γ-alloys. This observation could lead to isothermal kinetical dependence of the early stages of the precipitation experiments. We also want to retry RT experiments on annealed samples.

Finally we want to thank the ILL for the beam time allowed, and our local contacts for their great disponibility.