<table>
<tr><td>ESRF</td><td>Experiment title:
In-situ X-Ray Diffraction measurement of the evolution of phase fractions in W-doped TiAl alloys.</td><td>Experiment number:
MA - 3640</td></tr>
<tr><td>Beamline:
ID11</td><td>Date of experiment:
from: 14-02-2018 to: 16-02-2018</td><td rowspan="2">Date of report:
27-02-2018
Received at ESRF:</td></tr>
<tr><td>Shifts:
6</td><td>Local contact(s):
Dr. Pavel Sedmak</td></tr>
</table>

Names and affiliations of applicants (* indicates experimentalists):

Dr. Mikael Perrut *, Onera – The French Aerospace Lab, F-92322 Châtillon, France

Dr. Jean-Marc Joubert *, ICMPE (UMR 7182), CNRS, UPEC, F-94320 Thiais, France

Dr. Mathieu Fèvre *, Laboratoire d'Études des Microstructures, UMR 104 Onera-CNRS, ONERA BP72 92322 Chatillon Cedex, France

Thomas Vaubois*, Onera – The French Aerospace Lab, F-93222 Châtillon, France

Titanium-aluminides have recently been introduced on turbine blades of aero-engines such as the CFM-LEAP as replacement for the commonly used nickel-based superalloys. Their low-density and excellent specific properties, especially their creep resistance, enable the development of more complex and more efficient engines. However, due to their poor oxidation resistance at high temperatures, these alloys can only be used up to 700°C. One of the possibilities considered to improve their functioning temperature is to add refractory elements such as Niobium or Tungsten as alloying elements. However, despite a good knowledge of some ternary systems such as Ti-Al-Nb or Ti-Al-Cr, the Ti-Al-W ternary phase diagram is rather unknown. The purpose of our study is to explore this system in order to establish a new ternary assessment. In order to do so, we aimed to study *in-situ* the phase transformations in the near-TiAl composition range, with a specific interest for order/disorder transition such as the ordering of the β phase (BCC_A2) into β_0(BCC_B2). We report here our measurements performed at high temperatures (up to 1300°C) on various samples. These measurements were obtained on the beamline ID11 at the European Synchrotron Radiation Facility with a monochromatic beam of 69keV. The Debbye-Scherrer diffraction rings are registered by a 2D detector coupled to the ESRF FRELoN CCD-camera. The samples have a cylindrical shape (diameter between 1 and 2 mm, height ~1cm) and are glued on top of magnesia rods (diameter XXXX mm) with a high-temperature magnesia adhesive. The samples are placed in a tubular furnace with an internal diameter of 10mm and are protected from oxidation thanks to a constant flow of Argon. The furnace contains two Kapton windows, enabling the transmission of the beam. The temperature is measured via two thermocouples. The first one is located in the furnace about 1 cm above the sample which turned out to be not reliable while the second is glued at the basis of the sample. Despite a strong thermal gradient inside the sample itself due to the gas flow, the second thermocouple is much more accurate than the first one. In addition to this, complimentary DTA measurements are used to rescale the temperature properly. The furnace is mounted on a vertical translation stage, therefore enabling us to rapidly control its position regarding the sample.

During our 6 shifts at ID11, we had time to study six different samples. For the first experiment we performed, the results were not exploitable due to insufficient gas flow leading to a complete oxidation of the sample. For the second and third sample, the second thermocouple was not yet implemented thus preventing us to properly monitor the temperature of our sample, thus leading to the unexpected melting of the third sample. For the sixth sample, we encountered a problem with the glue as the sample fell at high temperature.

Overall, we performed a cycle of heating followed by controlled cooling on three different compositions, respectively $Ti_{68.75}Al_{25}W_{6.25}$, $Ti_{53}Al_{45}W_2$ and $Ti_{45}Al_{50}W_5$. Diffraction patterns were recorded both during heating and cooling. We studied the influence of sample preparation by using three different types of samples (as cast alloy, annealed and extruded).

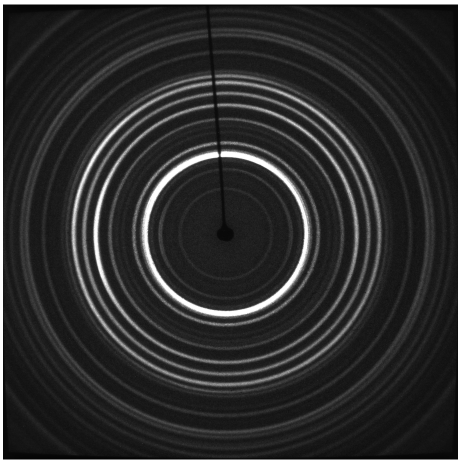

Figure 2: Diffraction pattern observed at room temperature for an extruded alloy (Ti_{53}-Al_{45}-W_2)

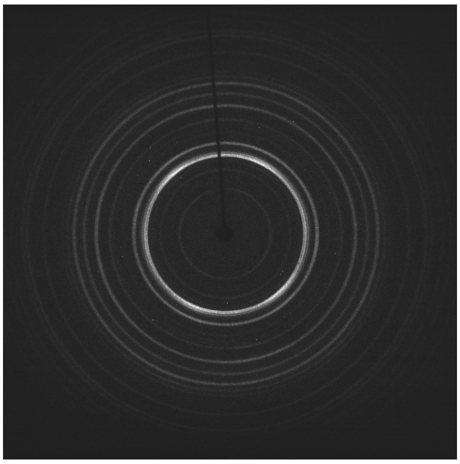

Figure 2: Diffraction pattern observed during heating (800°C) for an extruded alloy (Ti_{53}-Al_{45}-W_2)

As of now, only preliminary results are available, however some tendencies have been observed. Still, this report shall be updated in the upcoming weeks when more results are available.

So far we have observed two main tendencies/results:

- We have been able to observe the solvus of the gamma phase in the $TiAl_{45}W_2$ alloy.
- We have also observed the apparition of some signature peaks of the beta ordering on the near Ti3Al ($Ti_{68.75}Al_{25}W_{6.25}$) which were rather unexpected.

We plan on analyse our results over the next few weeks using Rietveld analysis

Conclusion

Altogether, these 6 shifts enabled us to gain a better understanding of phase transformations in the ternary system Ti-Al-W. However, due to the experimental issues we encountered, we were not able to study all the samples we meant to examine, especially the Ti-W binary alloy. Nevertheless, the first results we obtained are quite promising and further analyses are necessary. Overall, the beam time we benefited from was quite successful as it helped us discovering new aspects of phase transitions in our ternary system.