ESRF	**Experiment title: Microstructural characterization and phases transformation identification of highly complex self-fluxing alloy NiCrBSi obtained via low and high cooling rates**	**Experiment number**: MA-5643
Beamline: ID22	**Date of experiment**: from: 09/03/2023 to: 11/03/2023	**Date of report**: 24/05/2023
Shifts: 7	**Local contact(s)**: Andrew Fitch	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Joël Alexis (ENIT-LGP)

Olivier Dalverny (ENIT-LGP)

Yannick Balcaen* (ENIT-LGP)

Morgano Mokhtari (ENIT-LGP)

Anthony Ty* (ENIT-LGP)

Report:

As mentioned in the proposal, the two objectives of this experiment were to :

- Identify complex phases of NiCrBSi alloy obtained by L-PBF
- Establishing the equilibrium phases transformation sequence of NiCrBSi alloy

Phase identification

According to the literature and our investigations, we suppose that the equilibrium structure of NiCrBSi alloy is composed of the Ni-rich matrix, CrB, Cr_7C_3, Ni_3B, Ni_2Si and Ni_3Si. However, the microstructure of NiCrBSi produced by L-PBF is not described yet in the literature. Even with our investigations, nanometric crystals are still unidentified. Therefore, we utilised the angular resolution available at ID22.

We studied the powder in order to obtain the diffraction patern of the raw material, a cast sample, 13 heat-treated cast samples, 9 L-PBF samples produced with different parameters and 13 heat-treated L-PBF samples. For all 14 cast samples, 5 measurements spaced out by 1 mm were required due to the heterogeneity. Those analyses were automatically performed during the night with the use of the robot arm.

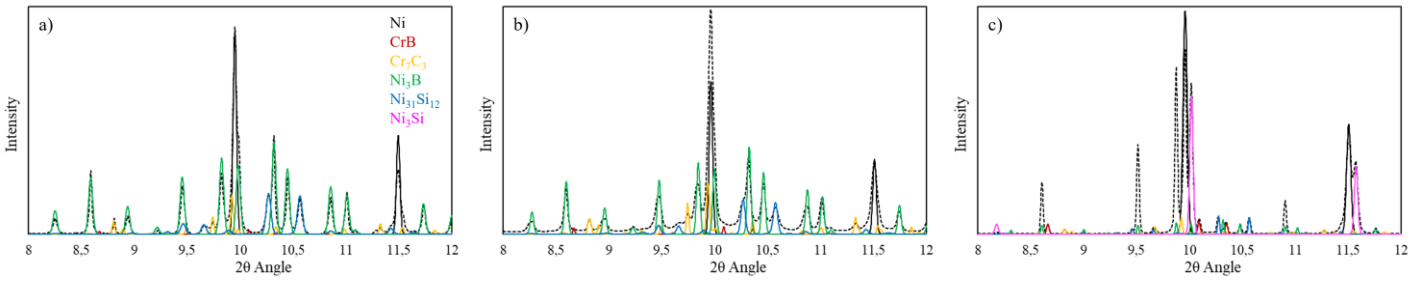

Fig.1 Phase identification of a) the powder b) the L-PBF sample c) the cast sample.

Compare to diffraction patterns previously obtained by conventionnal XRD, the angular resolution and the signal-to-noise ratio of those obtained at ID22 is far better. This angular resolution permit to separate intricated diffraction peak. Thus different phases were reliably identified. The powder and the L-PBF sample are composed by the Ni-rich matrix with CrB, Cr_7C_3, Ni_3B, and $Ni_{31}Si_{12}$ phases. The cast sample is composed by the same phases but with added Ni_3Si precipitates. It should be mentioned that cell parameters of those phases are slightly different compare with databases such as The Material Project (TMP), Crystallography Open Database (COD)

and Powder Diffraction File (PDF). Therefore, we followed advices of Jonathan Wright and created phases that match the diffraction pattern by tweaking cell parameters. The increase of preheating temperature on L-PBF samples does generate Ni_3Si precipitates. Furthermore, high temperature preheating and laser remelting supresse the $Ni_{31}Si_{12}$ phase. In a similar way, we studied the influence of heat treatment on the microstructure. This work is still under progress but the method is dialed. Those data obtained at ID22 concerning cell parameters will be completed by XRD-CT performed at ID11 and local analysis via TEM-SAED-EELS / EBSD.

Equilibrium phase transformation sequence

No *consensus* concerning the equilibrium and non-equilibrium transformation sequence of NiCrBSi alloys during the solidification has been found in the litterature. According to our investigations via DTA, several transformations might appear between 950 and 1050 °C. Therefore, *in situ*, synchrotron XRD was performed on the powder at ID22 in order to determine the nature of those transformations. The radiation furnace developped at ID22 was used. A heating and cooling rate of 15 and 100 °C.min^{-1} were successfully explored until 1200 °C. Phase identification was done similarly to previously described.

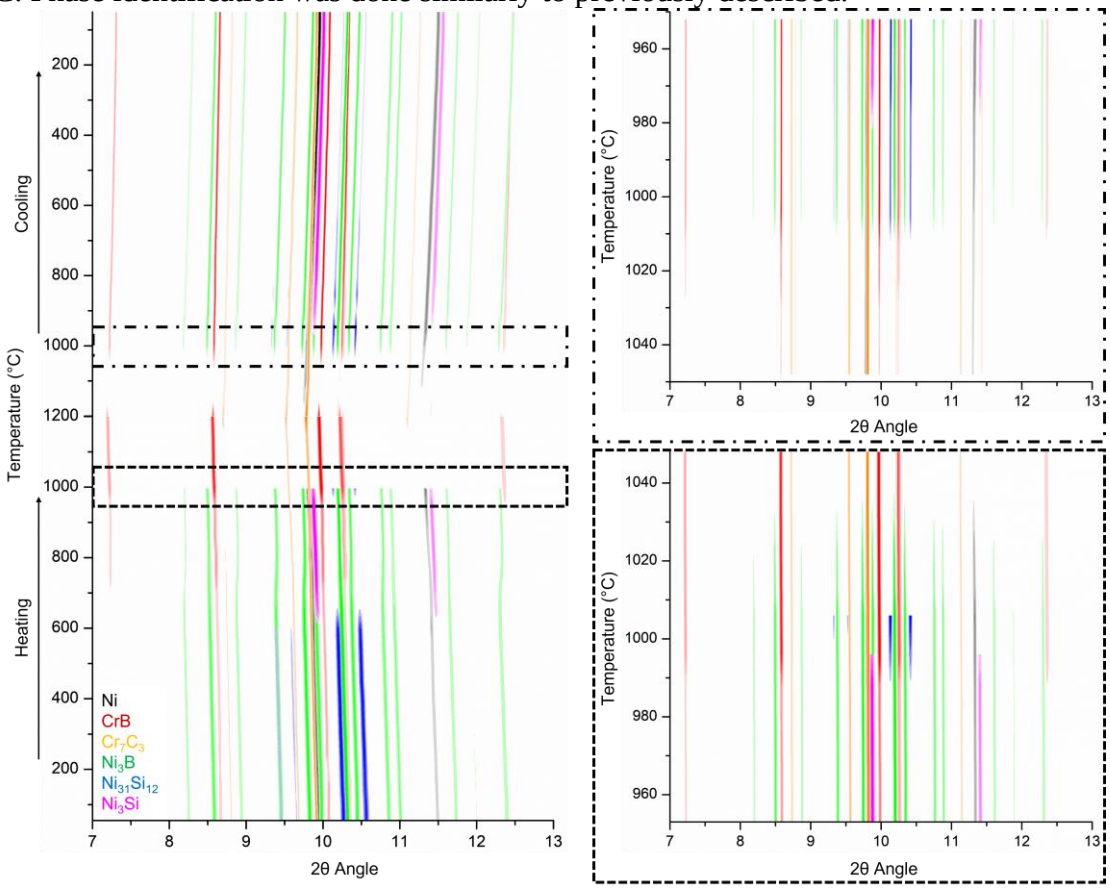

Fig. 2 In situ XRD analysis at 15 °C.min^{-1} on the powder.

During heating, $Ni_{31}Si_{12}$ peaks disappeares around 600 °C and Ni_3Si peaks appears at the same temperature. On the other hand, before melting, Ni_3Si peaks disappears and $Ni_{31}Si_{12}$ peaks re-appears. $Ni_{31}Si_{12}$ is the first phase to melt followed by Ni_3B, the Ni-rich matrix and CrB. During cooling, the Ni-rich matrix is the first phase to reappear at around 1100 °C followed by CrB, Ni_3B and $Ni_{31}Si_{12}$. Furthermore, Ni_3Si precipitates around 970 °C. A similar analysis for data obtained at 100 °C.min^{-1} and at 50 °C.min^{-1} on the L-PBF sample are still to be performed but the method is dialed.

Conclusion

The experiment was successfully carried on all samples. Phase identification was successfully carried and results match our expectations. Thus, they will be part of a paper concerning the microstructure of NiCrBSi produced by L-PBF and maybe another concerning heat treatments on those parts. *In situ* XRD analysis will be part of an article concerning equilibrium phase transformation of NiCrBSi alloy. Finally, those results will be part of my thesis concerning the understanding of NiCrBSi alloy.