Experimental report

25/01/2024

Proposal: **1-02-352**

Council: 10/2022

Title: In-situ strain monitoring during direct metal deposition of Inconel 718 by neutron diffraction

Research area: Materials

This proposal is a resubmission of 1-02-318

Main proposer: **Sandra CABEZA**

Experimental team: Alexander MEJIA REINOSO
Paula RODRIGUEZ
Volodymyr SVITLYK
Jacob Florian MAETJE
Rico HEMSCHIK
Franz MARQUARDT
Maximilian HEIDOWITZSCH

Local contacts: Thilo PIRLING
Sandra CABEZA

Samples: IN718 alloy: Ni-Fe-Cr with 5% Nb 2%Mo 1%Ti 0.5% Al

Instrument	Requested days	Allocated days	From	To
SALSA	7	6	11/05/2023	14/05/2023
			14/11/2023	19/11/2023

Abstract:

Throughout the AM process, complex thermal gradients, heterogeneous microstructure, and deep gradients of residual stresses (RS) within the bulk play a critical role for further mechanical solicitation of engineering (structural) components. Contrary to conventional techniques such as welding or casting, the strain evolution cannot be directly estimated by macroscopic measurements nor accurately predicted by computational modelling yet. Therefore, AM requires the implementation of new characterization approaches considering in-situ studies to disclose the controlling mechanisms and influence of fabrication parameters on phase and strain formation. Neutron diffraction at SALSA allows us to perform continuous monitoring in the bulk in shorter acquisition times thanks to the combination of high spatial resolution and flux2. This is a continuation from May2018 campaign and a resubmission from 2021 due to unavailable laser equipment at that time. In particular, we aim to investigate the effect of different energy inputs (from 0.025 ¿ 0.035 kJ/mm) with a similar setup and material at SALSA.

ILL - Experimental report

In-situ monitoring of Inconel 718 strain evolution during wire-DED

Experiment Nb. 1-02-352

Instrument	Local contacts	Beam times	Experimental team
SALSA	Sandra Cabeza Thilo Pirling	11.05.23-14.05.23 14.11.23-19.11.23	Maximilian Heidowitzsch Rico Hemschik Franz Marquardt Jacob-Florian Mätje

Overview: Wire direct energy deposition (DED) characterizes by rapid cooling and cyclic heating of deposited layers. Hence, dynamic thermal gradients that change over time and region within the bulk material are usually present causing repetitive liquid-solid and solid-state phase transformations. Consequently, strains comprising microstructural and residual stress (RS) contributions are present compromising the reliability and reproducibility of the process. A deeper understanding between process parameter, microstructure and RS is required to disclose the controlling mechanisms for process optimization. However, the microstructural and RS-based strain contributions are hard to decouple due to lack of detailed information about the overall microstructural evolution combined with in-situ stress states of the material. While laboratory scale methods do not possess the time and spatial resolution to disclose those mechanisms a combination of synchrotron and neutron characterization can yield new insights into that matter. In the framework of an ESA de-risk GSTP activity synchrotron characterization at ESRF-ID31 combined with neutron experiments at ILL-SALSA shall give insight in the stress and microstructural evolution during laser wire-DED.

A detailed analysis of the microstructural features during wire-DED synchrotron analysis was already successfully conducted at the ESRF-ID31 to disclose in-situ phase formation. A portable printing set-up was developed at IWS and employed for the consistent and reproducible fabrication and analysis of IN718 and IN625 wall specimens of 300 mm length, 2 mm thickness and 35 mm height. A single track unidirectional welding approach was chosen. Two different parameter sets were used to assess the effect of different energy inputs on the phase formation and evolution.

Sample name	Laser power in W	Welding speed in mm/min	Wire feed rate in mm/min	Working distance in mm	Focus shift in mm	Line energy in J/mm
Slow	550	500	0.75	9	-1.5	66
Fast	1100	1000	1.4	9	-1	57,6

Table 1: Processing parameters of the IN718 and IN625 straight wall samples.

The complete system set up is shown in Figure 1. The whole printing set up was located on an additional ESRF z-/y-axes system to achieve a relative movement between the beamline (x-ray beam) and deposited material. However, detector and beamline stayed at a fixed position at all times. A synchronized movement strategy of IWS and ESRF axes was elaborated and enabled the acquisition of valid in-situ data within the timeframe of a track length.

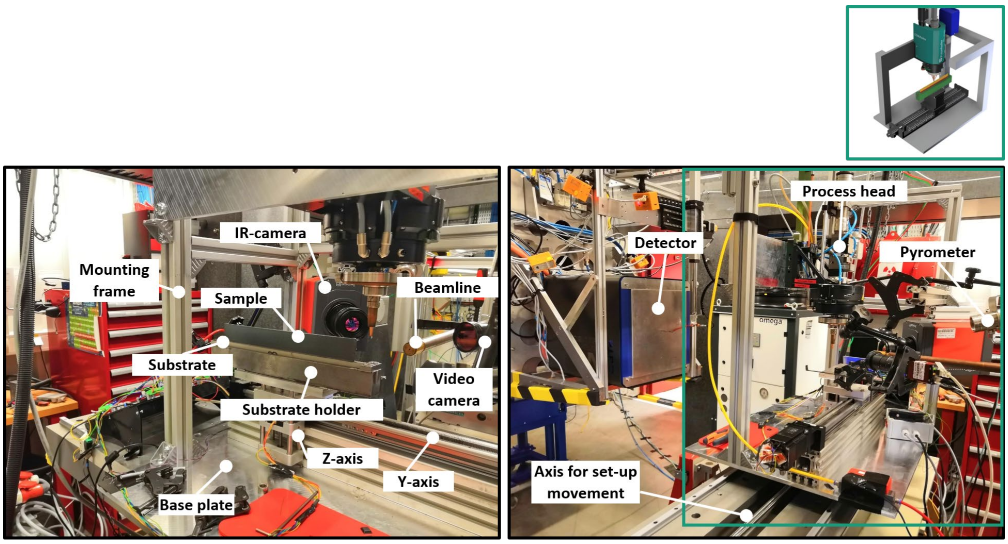

Figure 1: System set-up for in-situ synchrotron phase analysis at ESRF-ID31.

To get a comprehensive view on the overall stress states in the material neutron diffraction experiments are to be conducted during wire-DED and linked with the previous synchrotron measurements. In particular, it is aimed to investigate the effect of different energy inputs with a similar setup and material at SALSA.

ILL campaign 11.05.23-14.05.23: For the fabrication of specimens to be analysed by neutron diffraction the same system set-up as used at the ESRF-ID31 was employed. The set-up was located on the hexapod available at SALSA for flexible relative movement of fabricated samples and neutron beam. This way comparable and reproducible results can be ensured that later are coupled to the synchrotron data.
However, during first laser (Laserline LDM 2500-30) tests an inconsistent, sinusoidal laser power signal was observed. The cause of this issue could not be identified. Consultation with technicians of the laser supplier (Laserline) by phone did not yield any solutions. Since repairs of the laser are exclusively to be conducted by expert technicians of the supplier a fast and simple fix or laser repair onsite could not be realized. Experiments had to be cancelled since no safe and consistent fabrication of the samples could be ensured. Any fabrication and analysis of specimens would have yielded invalid data. Surprisingly, later laser tests at IWS showed no such laser power behavior. It was assumed that an unsuitable electric grit and power supply at ILL was causing the erratic laser signal. For later experiment campaigns a transformer was suggested to counteract that issue.
Rest beamtime was used for first ex-situ measurements on the specimens fabricated at ESRF (see Figure 1a). These measurements will act as reference and calibration for the later generated in-situ data. From each of the IN718 and IN625 a "slow" and "fast" fabricated specimen was analyzed. The walls were scanned along the build height in the middle of each sample (see Figure 1a, bottom left). Three strain components (see Figure 1b) were measured in each sample.

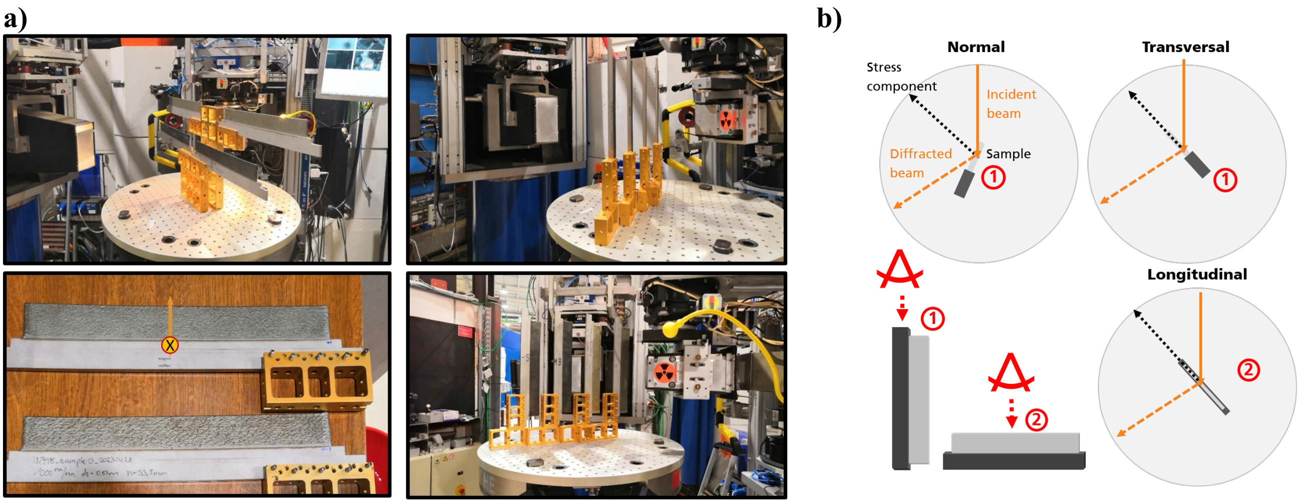

Figure 2: Ex-situ neutron diffraction set-up (a) and strain components measured at each sample (b). From each of the IN625 and In718 wall one "slow" and one "fast" sample was analysed.

ILL campaign 14.11.23-19.11.23: This campaign served as replacement of the beamtime in May 2023. Again, in-situ measuring campaigns were supposed to take place. In preparation of the experiments and to ensure laser functionality, this time, preliminary laser tests took place at ILL-SALSA on 11.10.23. A transformer with a power generator was used as power supply for the laser to eliminate the erratic laser power signal. First laser trials yielded success at that time and a continuous stable laser signal was achieved. It was assumed that the use of a transformer will solve the problems encountered during the campaign in May. However, after the set up of the system for the actual in-situ campaign and during first welding trials, the lasers fuses went off on laser restart. This issue started to accumulate until on every laser power-up the fuses of the power supply would switch off. On site attempts to fix the issue or repair the laser yielded no success. The beamtime had to be cancelled. A laser test at the IWS showed the same problematic. An inspection of the laser by Laserline technicians revealed that the power supply was defective. This might be due to laser transport.
To ensure success in future attempts of in-situ neutron diffraction during wire DED and to guarantee the functionality and integrity of the whole system, it is suggested to account for a preparation time of ca. 1 week before the actual beamtime e.g. before the start of a new reactor cycle. This will give enough time for unforeseen troubleshooting activities. Simultaneously the actual beamtime slot can be reduced to a minimum.