In-situ X-ray synchrotron measurements of Ni-based superalloys during electron beam powder bed fusion-manufacturing

INDUSTRIAL DEVELOPMENT PROJECTS

THE INDUSTRIAL CHALLENGE

Electron beam powder bed fusion (PBF-EB) is a promising additive manufacturing (AM) method for Ni-based superalloys because it enables complex shapes with low material waste. Compared with laser powder bed fusion (PBF-LB), its higher build temperatures and lower thermal gradients may also reduce the cracking that occur during PBF-LB and cause failure. However, in precipitation-strengthened γ/γ' Ni-based alloys, the intrinsic heat treatment during PBF-EB may still lead to formation of γ' which in turn influences manufacturability. Assessing PBF-EB for industrial use therefore requires a deeper understanding of microstructural evolution during processing.

WHY USING A LARGE SCALE FACILITY

The nm-sized γ' precipitates can be observed by transmission electron microscopy or bench-top/lab small angle X-ray scattering. They can even be observed using scanning electron microscopy (SEM) if they are large enough. However, due to the complex thermal history of the process, it is not clear when in the process they are formed. Furthermore, the rapid heating and cooling rates make it impossible to observe the rapid phase transformations with laboratory equipment. Only in-situ synchrotron experiments with small- and wide-angle X-ray scattering (SAXS/WAXS) and X-ray diffraction (XRD) provide the necessary temporal and spatial resolutions to reveal the formation of precipitates. This, however, also requires a custom-made PBF-EB machine with an unconfined powder bed and hence the same thermal conditions as the full-scale process.

HOW THE WORK WAS DONE

A custom-made PBF-EB machine, the 'MiniMelt', has previously been built and adjusted for the P21.2 and P61A beamlines at Petra III. Within this project, two beamtimes were allocated at P21.2. Cubic samples of two Ni-based alloys of different chemistry (representing different equilibrium fractions and temperature stabilities of γ') were build with MiniMelt at various build temperatures, electron beam powers and scan speed, resulting varying energy input. The powder bed in MiniMelt has a small hill, allowing measurements in transmission mode. A combined SAXS/WAXS setup focused on the formation of precipitates, while the evolution of strains was studied separately with an XRD setup. Both setups used a photon energy of 68 keV and acquisition rates up to 200 Hz. The beamline scientist Dr. Z. Hegedüs is acknowledged for support.

THE RESULTS AND EXPECTED IMPACT

The SAXS/WAXS data revealed microstructural evolution in the two Ni-alloys during the PBF-EB process. In the alloy with a chemistry representing a low γ' fraction, stable at lower temperatures, no precipitation was observed. However, in the alloy representing a higher fraction γ', stable up to higher temperatures, precipitates formed rapidly during cooling subsequent to melting (Figure) and coarsened during post-heating. Larger energy input resulted in larger particles and a higher depth in which newly formed particles can be found. Ex-situ SEM showed no strain age cracking, which is often reported after PBF-LB processing of these alloys. Together, these experiments propose that PBF-EB has the potential be suitable for industrial production of precipitation strengthened γ/γ' Ni superalloys, and motivates further work to develop optimized alloy compositions for PBF-EB.

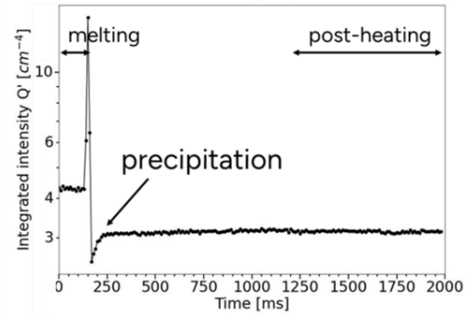

Figure. Integrated intensity Q' obtained from the SAXS signal indicates rapid precipitation during the cooling to build temperature after melting. Precipitation is assumed to start as soon as the temperature is below the solvus temperature of γ'.

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