In Situ Structural Analysis of $AlSi_{10}Mg$ for Additive Manufacturing – from Powder to Thermally Treated Parts

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Introduction

Printing 3-D, robust metallic structures via laser beam melting of alloy powders is a rapidly growing industry branch. Manufacturers of such parts optimize their processes to improve material properties, as well as to enhance the interchangeability of building platforms and thus, their economic flexibility. The number of critical parameters for 3-D printing is large and most simulations or macroscopic tests do not sufficiently predict the outcome of a recipe. Parts from the same powder alloys with slightly different building parameters do not possess the same mechanical properties due to the grade of intrinsic thermal treatment they experience in the respective laser-melting process. Differential scanning calorimetry (DSC) and X-ray diffraction are prominent techniques used to provide information on transitions and crystallinity in the material before and after additional treatments, but the results are often inconclusive with respect to morphological changes. Through ***in situ*** **heating experiments in TEM, applying EDXS and EELS** for structural and elemental analysis, we bridge this gap by studying the micro- and nanostructure of $AlSi_{10}Mg$ – a high-hardness lightweight alloy with well-known casting properties that is of great interest for additive manufacturing [1].

Materials & Measurements

Alloys for 3-D printing

- powders produced through gas atomization
- single powder beads with approx. 10 - 60 µm (diameter)
- in production, particles prone to very fast local cooling rates
- components (e.g. Si in $AlSi_{10}Mg$) “frozen” in an amorphous state [2, 3]

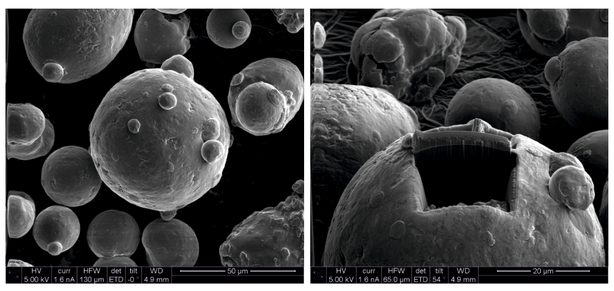

Figure 1. Left: $AlSi_{10}Mg$ power grains. Right: a FIB lamella is cut from a powder grain.

Samples

- **powder**: cut from a large powder grain (figure 1)
- **as-built**: cut from a melt pool identified via SEM
- **thermally treated**: reference for *in situ* thermal treatment

Comparability

- Micro- and nanostructure and morphological changes with temperature are very similar for powder and printed samples during *in situ* heating experiments in TEM.
- Thermally treated parts and in situ heated as-built samples exhibit corresponding features.

In situ thermal treatment of an as-built printed part

- The eutectic silicon network in the as-built specimen is stable in its granular, coral-like state up to 160 °C. Building stresses and nano-grains govern the mechanical properties and high yield stress of the material.
- At temperatures of 200 °C and higher, the Si network visibly starts to rearrange (figure 2).
- From 280 °C onwards, Si forms globular crystalline precipitates with diameters of about 100 to 200 nm. Small fractions of the original Si network remain at sub-grain boundaries of the α-Al matrix (figures 3, 4).
- Mg accumulates at interfaces.
- Results back up macroscopic measurements (exothermic DSC signals, linked to crystallization of Si, figure 4) for thermally treated samples [1, 3, 4].

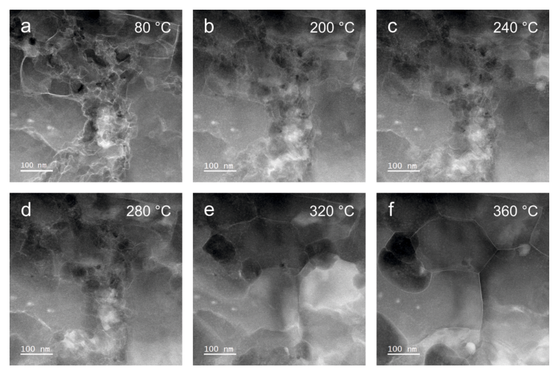

Figure 2. Evolution of a eutectic Si network in $AlSi_{10}Mg$ from the 3-D printed as-built state to ordered particles through an *in situ* heating experiment. a) 80 °C b-f) 200 °C - 360 °C (STEM, HAADF).

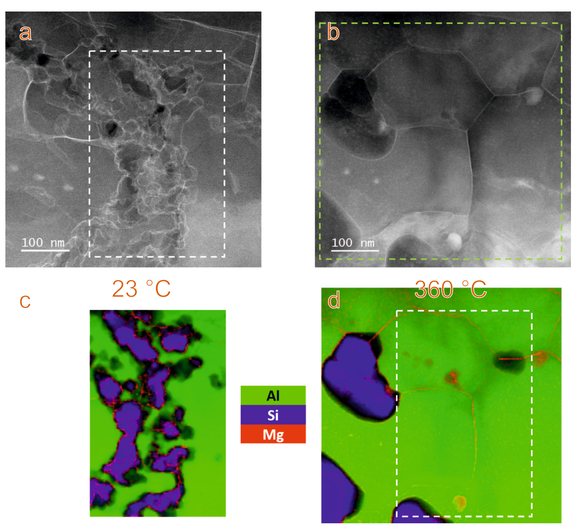

Figure 3. Elemental distribution of Si (blue) and Mg (red) in the Al (green) matrix acquired through STEM-EELS in an *in situ* heating experiment of an $AlSi_{10}Mg$ as-built specimen without prior thermal treatment. a, c) at 23 °C; b, d) at 360 °C. The elemental map (c) is taken from the dashed white area in (a). The elemental map (d) is taken from the region in (b), bordered in dashed green and includes (c) (dashed white).

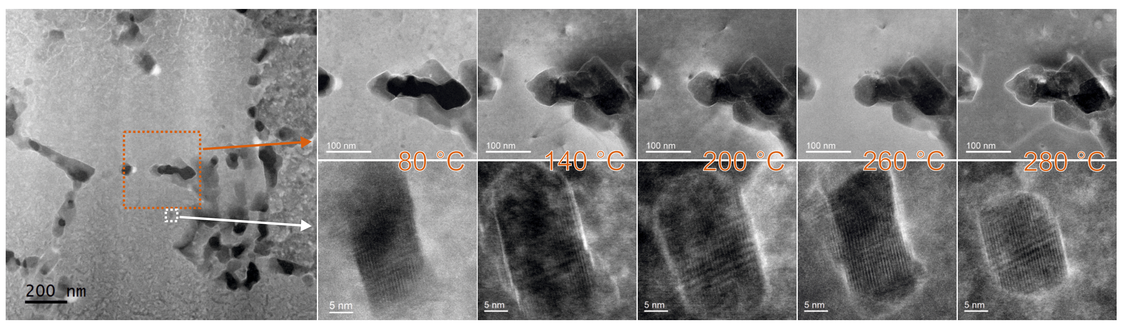

Figure 4. Left: overview at lower magnification; HAADF STEM image indicating exemplary regions followed during *in situ* heating. Top row: HAADF images of eutectic Si particles in the as-built printed sample at different temperatures. Bottom row: progressing crystallization in atomic resolution images of a Si nanoparticle in the Al matrix.

Conclusion

- *In situ* heating experiments in TEM provide a unique insight into material transitions at the micro- and nanoscale.
- The apparent modifications in $AlSi_{10}Mg$ significantly alter the strength of materials and manufacturers can therefore trim their building process parameters to either reach higher hardness or ductility in printed alloys.
- In the presented experiments we comprehensively show that thermal treatment at temperatures higher than 280 °C rearranges the microstructure from a production-induced disordered (and thus, macroscopically hard) “frozen” state to a predominant α-Al-matrix with crystalline Si precipitates and Mg in interfacial regions.

References & Acknowledgement

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