Dislocation network in additive manufactured steel breaks strength–ductility trade-off

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Most mechanisms used for strengthening crystalline materials, e.g. introducing crystalline interfaces, lead to the reduction of ductility. An additive manufacturing process – selective laser melting breaks this trade-off by introducing dislocation network, which produces a stainless steel with both significantly enhanced strength and ductility. Systematic electron microscopy characterization reveals that the pre-existing dislocation network, which maintains its configuration during the entire plastic deformation, is an ideal "modulator" that is able to slow down but not entirely block the dislocation motion. It also promotes the formation of a high density of nano-twins during plastic deformation. This finding paves the way for developing high performance metals by tailoring the microstructure through additive manufacturing processes.

Introduction

The motion of dislocations governs the plastic deformation, hence the mechanical properties of many metals [1–3]. The strength of the metals can be improved by hindering dislocation motion through the designing of microstructure including introducing secondary phases, grain boundaries, and other internal interfaces [4]. Unfortunately most of such strategies that effectively strengthen materials sacrifice ductility, resulting in the so called strength–ductility trade-off [5]. Although a few methods have shown the capability of improving strength while retaining the ductility of materials (for instance by introducing coherent twin boundaries [2,6], introducing bimodal grain sizes [7] and by controlling the size, morphology and distribution of secondary phases [8,9]), making final parts with complex shapes from these methods requires intensive additional machining and may even not be feasible in some cases.

Selective laser melting (SLM) is a type of additive manufacturing (AM) processes which is now rapidly changing the ecosystem of manufacturing by enabling the manufacturing of complex components directly from digital files, thus benefiting the customized production and the freedom of designing [10]. During SLM, particle granules are fused directly into 3D components by repetitive scanning of a high energy laser beam over each layer of powder granules, thereby consolidating them via partial or full melting. Another important feature of AM is the ultrafast cooling rate (10^3–10^8 K/s). Unlike the other rapid cooling techniques e.g. splat quenching and melt spinning which can produce only metals in low dimensional shapes e.g. metal powder, ribbon and foil, AM can produce metals in 3-dimensional shapes (bulk parts) with an extraordinarily high cooling rate [11–14]. The bulk metal parts show microstructures distinct from those produced by traditional manufacturing routes such as casting and wrought processes [15–21]. In this study, we show that a dislocation network structure with the accompanying segregation of the alloying elements produced during SLM manufacturing of 316L stainless steel (316LSS) leads to unprecedented mechanical properties of a combination of enhanced yield strength and

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ductility compared to those with the same composition but produced in the other manufacturing processes [22–28]. In-situ SEM and TEM study reveals that the dislocation network with the accompanying segregation provides a high density of "flexible interfaces" that significantly tunes the dislocation behaviors, resulting in the ameliorated mechanical properties. The results indicate the possibility to directly manufacture products with a good combination of strength and ductility while retaining the benefits of the process in manufacturing parts with complex or customized geometries.

Materials and methods

Sample manufacturing process

As received gas-atomized spherical 316LSS powder with granular sizes ranging from 10 to 45 μm was purchased from Carpenter powder products AB, Torshälla, Sweden. The standard build was performed by a selective laser melting facility EOSINT M270 (EOS GmbH, Krailling, Germany) equipped with a continuous Nd:YAG fiber laser generator with maximum 200 W power output and typically 70-μm diameter laser spot. During the building process, a layer of powder (20 μm in thickness) was laid by a recoating blade on a steel building plate which was preheated to 80 °C. The full laser power of 200 W was used and the laser beam was moving at the speed of 850 mm/s. The laser scanned line by line along the same direction at the same layer and with the line spacing of 100 μm. After the scanning was complete, a new layer of powder was laid and the laser scanned the new layer with the scanning direction rotated by 67°. The sample was built up by repeating this process.

To investigate the effect of scanning speed on dislocation cell size, the samples were built up using standard parameters and the last layer of each sample was scanned by laser with different scanning speeds and line spacings (7000 mm/s, 10 μm; 4250 mm/s, 20 μm; 283 mm/s, 300 μm). The corresponding SEM images were taken from the area within the top layers.

Tensile tests

Tensile test specimens (as-build size Φ8 × 52 mm) were prepared by SLM using standard building parameters and machined to cylindrical test specimens (Gage length: 12 mm; gage diameter: 3 mm). All the tensile test bars were built in the same build and with the longitudinal axes along the building direction. Tensile tests were performed according to ASTM E8 with a strain rate of 0.015 min^{-1} up to yield point, and afterward 0.05 min^{-1} till failure. An extensometer was used to measure the elongation. The reported values in this study for tensile properties were the average values of 5 tests.

Micropillar tests

For the micropillar compression test, two pellets were cut from the same bar built with the longitudinal axis along the building direction. One of them was packed in the stainless steel envelop and heated to 1050 °C with the ramp rate of 10 °C/min, kept for 2 h and followed by water quench. The other one was kept in the as-SLMed state. Two pellets were ground and polished before micropillar experiment. A commercial Hysitron PI85 PicoIndenter installed inside a Tescan Mira-3 scanning electron microscope was used for micropillar compressions. The micropillars with a diameter of about 5 μm, length of about 10 μm and tapering angle less than 5 degree were fabricated in a FEI Quanta 3D FEG Focus Ion Beam (FIB) by Ga^+ ion beam with the current ranging from 30 nA to 0.1 nA at 30 kV. Both of the two micropillars were fabricated from the grains with the (056) plane parallel to the top surface. The micropillars were then compressed using a flat punch diamond tip with a diameter of 20 μm and a constant loading rate of 100 $\mu N/s^{-1}$.

TEM analysis

TEM specimens were twin-jet electropolished in an alcoholic solution containing 5 vol.% perchloric acid at 30 mA and −25 °C. Equipped with both bright field and annular dark field detectors, a Cs-corrected FEI 80-200 G^2 with Super-X operated at 200 kV is employed to analyze the microstructure and elemental distribution of the SLMed 316LSS. The in-situ tensile tests were achieved by a Gatan model 654 single-tilt straining holder in a FEI Tecnai G2 F20 TEM operated at 200 kV.

SEM images were taken on the etched surfaces. Etching was done by submerging the mechanical polished samples into the etching agent ($HF:HNO_3:H_2O$ = 1:4:45) for 60 s.

Results and discussion

Tensile properties of the SLMed 316LSS and TEM characterization of the dislocation network structure

Figure 1b shows a component with the dimensions of 28 cm × 16 cm × 16 cm and a built-in complex internal cooling channel system manufactured using SLM process from 316LSS powders (particle size: 10 –45 μm) for the potential application as the first wall panel part in the International Thermonuclear Experimental Reactor (ITER). Tensile tests reveal that the SLMed 316LSS shows notable improvement in both strength and ductility compared to the fully dense 316LSS processed by the other manufacturing methods (Figure 1a) [22–28]. The tensile yield strength of 552 ± 4 MPa and elongation to failure of 83.2 ± 0.7%, was obtained for the SLMed 316LSS (along the building direction). In contrast, the wrought-annealed 316LSS with an average grain size of 17.5 μm from Ref. [22] shows yield strength of 244 MPa and failure elongation of 63% [22]. A number of previous research on SLMed 316L reported that the process improves the yield strength but reduces or has little effect on ductility [11,29,30]. The ductility of metals is sensitive to the defects like voids and cracks whose presence largely depends on the process parameters. Only when the defects are suppressed, the contribution from the other factors would be revealed.

Residual stress can be generated during SLM process, but it was not considered as the major factor affecting the tensile results in this work. Previous studies show that residual stress in SLMed sample can be comparable to the yield strength of the material near the top surface but is much lower in the lower part of the sample [31–33]. The gage section of the tensile test bar in this study is far below the top surface. Moreover the building plate was preheated to 80 °C during the process to reduce residual stress. The microstructure of the material is then considered as the main reason for the ameliorated mechanical properties. The SLMed 316LSS is composed of mainly columnar grains with diameters ranging from a few to tens of micrometers and lengths up to hundreds of micrometers. TEM analysis reveals a unique

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