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Neutron diffraction measurements of residual stress in additively manufactured stainless steel

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ABSTRACT

Charpy test specimens were additively manufactured (AM) on a single stainless steel plate from a 17–4 class stainless steel using a powder-bed, laser melting technique on an EOS M280 direct metal laser sintering (DMLS) machine. Cross-hatched mesh support structures for the Charpy test specimens were varied in strut width and density to parametrically study their influence on the build stability and accuracy as the DMLS process has been known to generate parts with large amounts of residual stress. Neutron diffraction was used to profile the residual stresses in several of the AM samples before and after the samples were removed from the support structure for the purpose of determining residual stresses. The residual stresses were found to depend very little on the properties of the support structure over the limited range studied here. The largest stress component was in the long direction of each of the samples studied and was roughly 2/3 of the yield stress of the material. The stress field was altered considerably when the specimen was removed from the support structure. It was noted in this study that a single Charpy specimen developed a significant tear between the growth plate and support structure. The presence of the tear in the support structure strongly affected the observed stress field: the asymmetric tear resulted in a significantly asymmetric stress field that propagated through removal of the sample from the base plate. The altered final residual stress state of the sample as well as its observed final shape indicates that the tear initiated during the build and developed without disrupting the fabrication process, suggesting a need for *in-situ* monitoring.

1. Introduction

Additive manufacture (AM) refers to the process of "growing" parts to near net-shape using a bottom-up methodology through deposition of material from either powder or wire feed. This is in contrast to the more traditional top-down fabrication methodology that involves subtractive techniques where material is removed from cast or wrought billets to achieve a particular geometry. Powder-bed AM processes for metals, in particular, often exhibit strong thermal gradients and rapid quenching of the deposited material. These necessarily result in large, often yield-level, residual stresses [1] in the as-deposited part which can result in premature fracture if the part remains on the build substrate or large-scale distortions when removed from the substrate.

The use of support structures during powder-bed based metal AM fabrication is ubiquitous. The support structure is used to control heat transfer between the part and the base plate and constrain the part during manufacturing. The support is a hatched porous structure, where the hatching parameters (cell size and porosity) are considered important manufacturing parameters affecting the residual stresses. After fabrication has finished, the support structure and base plate are removed from the final part through subtractive machining techniques. Due to the expense of feedstock material and the time associated with fabrication, studies have focused on the minimum density needed for a successful support structure [2,3]. In addition to looking at density, other studies have focused on the shapes and spacing of the struts that form support structures [4,5] These studies have generally focused on geometric stability of the final part after removal of the support structure and base plate. No study, however, has quantified measures of the stress state within parts prior to and after removal from the base plate as a function of support structure.

The obvious need has motivated several residual stress measurements in additively manufactured samples using, for instance mechanical relaxation techniques (*e.g.* crack-compliance [1]) as well as x-ray [1,6,7] and neutron diffraction techniques [8–11]. Neutron diffraction is particularly relevant because neutrons penetrate bulk distances (cm's) into most structural materials [12], *e.g.* steel, copper, nickel, *etc*, allowing for non-destructive mapping of multiple stress components at depth in an AM part. Moreover, the non-destructive nature of neutron diffraction allows for evolutionary measurements of residual stress in the same part after multiple processing steps, e.g [13], such as

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before and after an AM part is removed from the substrate, or before and after hot-isostatic pressing. The advent of high energy synchrotron x-ray sources (>60 keV), such as the 1ID beamline [14] at the Advanced Photon Source (APS), also offers the ability to non-destructively map stresses at depth. However, due to the low diffraction angle associated with high energy x-ray diffraction, it is often difficult to determine through-thickness stress components in 3-dimensional parts and one must often extrapolate, resulting in increased uncertainty [15]. A drawback of both neutron and synchrotron based diffraction measurements of residual stress is that access to the required instrumentation is extremely limited. Thus, the best usage of the limited access to the relevant beamlines is to couple the residual stress measurements with computational modeling (often finite element analysis) to provide validation of the model, which can then be used to predict and optimize the final properties (*e.g.* residual stress) in parts as a function of input parameters.

In this study, several samples with a Charpy test geometry [16] were additively manufactured on a solid base plate with mesh support structures varied in order to control the conduction of the heat and the mechanical constraint of the sample. The Charpy test specimen geometry was convenient and added a feature (the notch, grown perpendicular to the base plate) which could potentially alter the residual stress profile. Neutron diffraction was used to profile the lattice parameter of the AM samples with spatial resolution both before and after removal of the samples from the sub-structure. The residual stresses were determined from the variation of the observed lattice parameter from a reference value.

2. Experimental

2.1. Sample preparation

The thickness of the part and mostly uniform cross sectional area make the Charpy specimen geometry suitable for neutron-based scoping experiments on thick z-axis parts. 10 mm tall rectilinear block style support structures were generated as separate STL (standard tessellation language) files consisting of cell hatchings ranging from 0.25 to 0.6 mm and populating 0.3 mm fragmentations in increments ranging from 3 mm to 5 mm. The support height and other fixed variables were determined *via* a down-selection process based on several preliminary tests exploring upper and lower bounds for key parameters.

Overall, 14 samples with Charpy geometry (labeled A-N) were grown on a single square stainless steel plate (252 mmx252 mm) for multiple purposes, including these residual stress measurements. Fig. 1(a) shows a schematic of the build plate, Fig. 1(b) a schematic of an individual sample, including the coordinate system used in this paper. The specimens were grown in a single build using a powder-bed, laser melting technique on an EOS M280 direct metal laser sintering (DMLS) machine. The hatching (h) and fragmentation (f) of the support structure were varied across the 14 samples in a controlled manner to monitor their influence on the final sample. The base plate was sectioned between individual specimens to allow passage of the neutron beam (neighbor specimen would have interfered). After completion of residual stress measurements on the as-built specimens, they were removed from the base plate and mesh sub-structure using a cut off wheel and a subset of the residual stress measurements were repeated on the free specimens.

Table 1 lists different parameters which were varied in the growth of the samples studied here-in. Neutron diffraction based residual stress measurements were completed on four of the Charpy samples; A, C, D, and K. Pictures of Samples A and C mounted in the diffractometer are shown in Fig. 2(a) and (b) Samples A and C were constructed with the same support structure parameters, h=0.25 and f=3, while D and K had distinct parameters, h=0.25, f=5 and h=0.35 and f=4, respectively. Sample A was intentionally grown closer to the edge of the plate than is typically done, 26.7 mm from the edge, while C was grown closer to the center, 80.2 mm from the edge, to study the effect of the altered heat flow at the edge..

Significant separation of the support structure of Sample A from the build plate was observed, see Fig. 2(b) The tear occurred on the side of the sample nearest the lateral edge of the plate and furthest along the direction of travel of the recoat blade as highlighted in Fig. 1(a) This single observation in Sample A is not sufficient to concluded that the growth position near the top of the plate, which alters the heat flow, is the definitive cause of the tear between the growth plate and mesh sub-structure. A stochastically occurring defect could equally well have been the cause. However, we note that Sample H (not studied further here-in) was the only other sample to manifest such a tear and it was grown in a position symmetrical to A.

A 3.5 mm cube was cut from Sample J, made with parameters identical to A and C, to be used as a reference lattice parameter, a_0, measurement. Compression and tension samples were EDM'ed from two of the samples, B and J to determine the macroscopic strength properties of the as-deposited material. The compression specimen was a cylinder 6.3 mm in diameter by 15 mm long. The tension sample was a threaded end specimen, ASTM E8 – 04 (Sub-size Round Tension Test Specimen) with a 3 mm diameter in the gauge length.

A FARO Edge HD Laser Line Probe was used to scan the surface geometry of sample M before and after removal from the base plate in

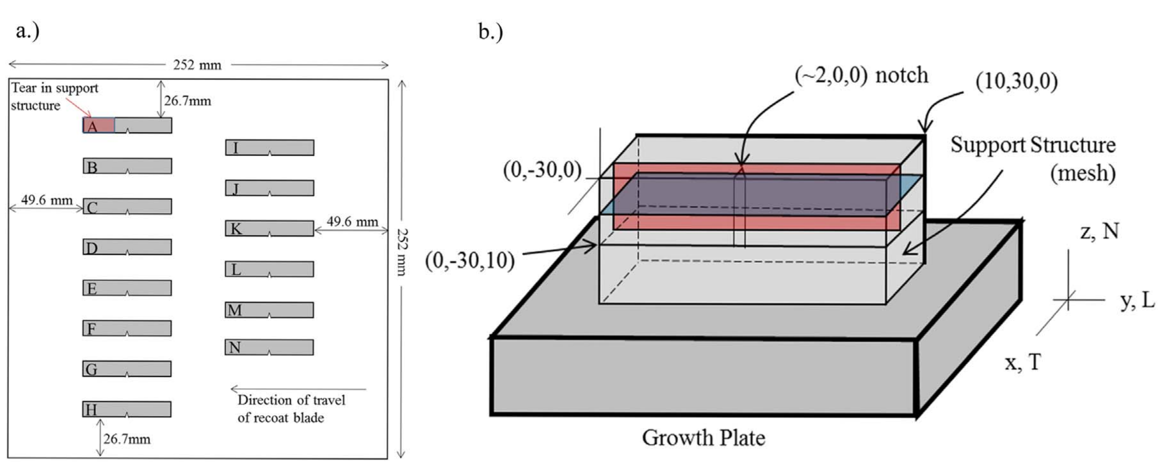

Fig. 1. (a) Schematic (roughly to scale) of build with 14 Charpy specimen. (b) Schematic of individual sample showing coordinate system and measurement loci discussed in the text. All units in mm. The origin (0,0,0) is at the top of the front face at the center length as shown, putting the tip of the notch at roughly (2, 0, 0) mm. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)