Neutron Characterization for Additive Manufacturing

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Nondestructive examination of complex additive manufactured components using neutrons is a valuable technique for imaging and measuring residual stress.

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Oak Ridge National Laboratory (ORNL) is leveraging decades of experience in neutron characterization of advanced materials together with resources such as the Spallation Neutron Source (SNS) and the High Flux Isotope Reactor (HFIR) shown in Fig. 1 to solve challenging problems in additive manufacturing (AM). Additive manufacturing, or three-dimensional (3-D) printing, is a rapidly maturing technology wherein components are built by selectively adding feedstock material at locations specified by a computer model. The majority of these technologies use thermally driven phase change mechanisms to convert the feedstock into functioning material. As the molten material cools and solidifies, the component is subjected to significant thermal gradients, generating significant internal stresses throughout the part (Fig. 2).

As layers are added, inherent residual stresses cause warping and distortions that lead to geometrical differences between the final part and the original computer generated design. This effect also limits geometries that can be fabricated using AM, such as thin-walled, high-aspect-ratio, and overhanging structures. Distortion may be minimized by intelligent toolpath planning or strategic placement of support structures, but these approaches are not well understood and often "Edisonian" in nature.

Residual stresses can also impact component performance during operation. For example, in a thermally cycled environment such as a high-pressure turbine engine, residual stresses can cause components to distort unpredictably. Different thermal treatments on as-fabricated AM components have been used to minimize residual stress, but components still retain a nonhomogeneous stress state and/or demonstrate a relaxation-derived geometric distortion.

Fig. 1 — *(left) Spallation Neutron Source (SNS); (right) High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory, Tenn.*

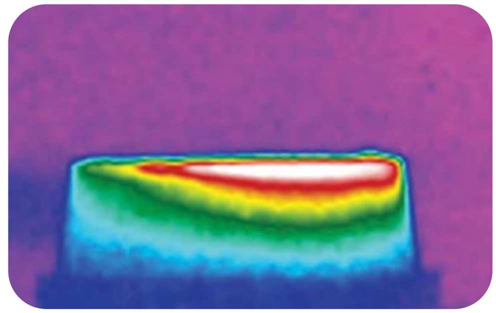

Fig. 2 — *Thermal image of a layered additive manufacturing process shows significant temperature gradients near the deposition zone*[1].

Industry, federal laboratory, and university collaboration is needed to address these challenges and enable the U.S. to compete in the global market. Work is currently being conducted on AM technologies at the ORNL Manufacturing Demonstration Facility (MDF) sponsored by the DOE's Advanced Manufacturing Office. The MDF is focusing on R&D of both metal and polymer AM pertaining to in-situ process monitoring and closed-loop controls; implementation of advanced materials in AM technologies; and demonstration, characterization, and optimization of next-generation technologies. ORNL is working directly with industry partners to leverage world-leading facilities in fields such as high performance computing, advanced materials characterization, and neutron sciences to solve fundamental challenges in advanced manufacturing. Specifically, MDF is leveraging two of the world's most advanced neutron facilities, the HFIR and SNS, to characterize additive manufactured components.

Why neutrons?

Neutron techniques might be less familiar than x-ray methods to materials scientists and engineers, but are similar in many ways. For example, x-ray and neutron diffraction both use *Bragg's Law* to describe scattering from single crystals, powders, and polycrystalline solids. However, one important difference is that neutrons are much more penetrating than x-rays in most materials. X-ray scattering occurs within a few microns (with lab sources) to a few millimeters (using high-energy synchrotron radiation) of the surface, whereas neutrons often penetrate several centimeters. Therefore, neutrons serve as a nondestructive probe of the bulk microstructure in engineering solids. Another difference is that x-ray scattering inten-

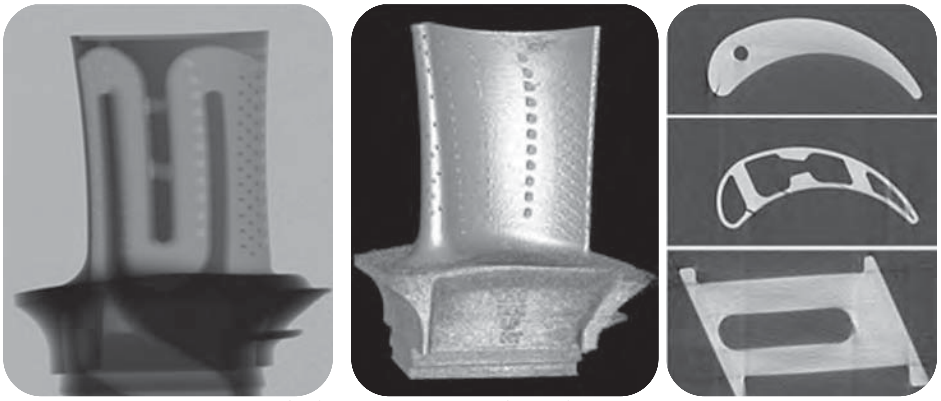

Fig. 3 — *Neutron radiograph (left), volume rendered (center), and transverse slices at top, middle, and bottom (right) of turbine blade fabricated using additive manufacturing. Blade height is ~76 mm.*

sity increases proportionally to the electron density of a material (or approximately to the atomic number Z). Consequently, light elements such as hydrogen, boron, and lithium make very little contribution to the overall scattering in the presence of heavier elements. In contrast, neutron scattering depends on the neutron scattering length, which is not a function of Z, and which may vary widely even for different isotopes of a particular element. Consequently, appreciable scattering signal can be obtained from light elements using neutrons.

While portable neutron sources exist, most material science and engineering work occurs at large, fixed neutron sources located at large national and international facilities[2]. Two source types are reactor and spallation. Reactor-based sources (e.g., HFIR) use fission to continuously generate a flux of neutrons, while spallation sources (e.g., SNS) generate neutrons by accelerating protons to high energies, which hit a target such as tungsten or mercury, causing emission of neutron pulses. The spallation process is analogous to a cue ball hitting a rack of balls on the break in billiards. Combining neutron source and detectors enables characterization of various aspects of materials.

Neutron computed tomography

Conventional two-dimensional (2-D) neutron radiography and three-dimensional (3-D) computed tomography are based on neutron beam attenuation through matter. Attenuation and contrast are directly related to total neutron cross section, which depends on neutron interactions with the atomic nucleus (e.g., coherent and incoherent scattering and absorption of neutrons). Neutron cross section does not depend on Z in a simple way: in many cases, a low-Z material is readily imaged despite the presence of surrounding high-Z material; not easily done using x-rays. Neutron cross sections are known, tabulated, and expressed in units of barn (1 barn = 10^{-24} cm^2). Neutron cross section can be thought of as the area of each nucleus as seen by the neutron[3] with larger cross sections being more attenuating. Attenuation coefficient can be measured at a continuous-source facility, such as a reactor-based neutron imaging beamline (HFIR).

Neutron computed tomography (nCT) reconstructs a 3-D image of the contrast due to attenuation of neutrons inside an object by transforming multiple 2-D radiographs of the object as it is rotated around its axis. Neutron CT was performed at HFIR's CG-1D cold neutron imaging prototype facility[4] on Inconel 718 turbine blades fabricated by additive manufacturing using direct laser metal sintering (DLMS) at Morris Technologies (Cincinnati, Ohio). Neutron radiographs are generated using a LiF/ZnS scintillator that converts neutrons into light, which is subsequently detected by a CCD camera. Spatial resolution at the sample position is approximately 75 μm. The radiograph and nCT data in Fig. 3 indicate a relatively low neutron attenuation through the blade itself, and higher attenuation at the base of the object. The dark/low transmission region is likely due to the increased thickness at the blade base. Some internal air/sample interfaces show an interesting texture that could affect airflow through the turbine. After reconstruction, the volume rendering provides a 3-D visualization of the object (Fig. 3 center). Quantitative analysis is often based on transverse slices obtained from the reconstructed piece (Fig. 3 right).

ORNL is developing a world-class neutron imaging instrument called VENUS (Versatile nEutron imagiNg instrUment at Sns), which will use the SNS in a unique way to measure and characterize large-scale and complex systems. VENUS will offer the opportunity to advance scientific research in a broad range of areas such as energy, materials, additive manufacturing, transportation, engineering, plant physiology, biology, and archeology[5]. It will have unprecedented spatial resolution (~10 μm), providing 3-D visualization of surface texture and porosity. The unique capabilities are derived from the intrinsic SNS time-of-flight (TOF) properties, which allow probing the wavelength-discrete neutron attenuation[6]. Energy-selective and Bragg edge-imaging capabilities will provide enhanced contrast mechanisms, material identification, and the opportunity to map strain inside samples.

Neutron residual-strain measurements

In addition to tomography, neutron diffraction can be used to nondestructively measure residual strain in a material and determine residual stresses. Strain is determined through *Bragg's Law* by measuring distances between crystallographic planes of the strained sample and a reference. Figure 4 illustrates how the sample interior is mapped. The nominal gauge volume is fixed in space on a goniometer and defined by the intersection of projections of incident and receiving slits. The sample is moved (x, y, and z, and rotation) relative to the gauge volume and mapped. While diffraction outside the gauge volume occurs along the beam path, it is not recorded because these neutrons are blocked by the receiving slits. The direction of the measured strain component is given by the scattering vector, which bisects the incident and diffracted beams and is perpendicular to the diffracting planes.

Characterization of EBF³ samples

NASA Langley Research Center (Hampton, Va.) has been working on large-scale metal additive manufacturing for the past ten years. The electron beam freeform fabrication process (EBF^3) uses a high-power electron beam with wire feedstock to fabricate large structural aerospace components. These components often exceed a meter in length and require many kilograms of deposited material. The incremental nature of the additive process combined with the large scale of EBF^3 fabricated components results in high levels of distortion and/or residual stress. Understanding how these residual stresses evolve is critical in developing effective mitigation strategies.

Two applications of primary interest for the EBF^3 platform are terrestrial-based manufacturing of aerospace structures and remote, space-based fabrication of hardware. For the former, knowledge of residual stress accumulation helps develop fixturing, path planning, and intermediate heat treatment strategies minimize throughput time and out-of-tolerance distortion. For the latter, knowledge of the residual stresses helps predict final bulk properties, as heat treatment may not be available for structures fabricated extraterrestrially.

NASA recently initiated neutron diffraction measurements on EBF^3-deposited aluminum samples using the Vulcan Engineering diffractometer at SNS. Initial measurements evaluated the impact of an intermediate thermal anneal treatment on residual stress. The stress relief heat treatment shifted the residual stresses from the substrate into the deposited material. This is important for the EBF^3 process where the substrate plate is usually incorporated into the final part and often accounts for the bulk of out-of-plane distortion. These data were used to develop basic models using commercially available finite element modeling software. Additional trials at SNS scheduled for early 2013 will help validate the early models and provide a predictive capability to minimize residual stress and distortion in large-scale additive structures.

Characterization of DLMS turbine blades

Experiments were also conducted on the VULCAN beam line to determine if residual strains could be measured in complex components with internal structure, which is also of interest elsewhere[8]. A series of turbine blades were fabricated from Inconel 718 using DLMS technology. The blades have a wall thickness down to 1 mm and curved internal cooling passages (see Fig. 3). Three blades were analyzed at VULCAN together with a strain-free reference powder (Fig. 5). Slits on both the incident and diffracted beams give a gauge volume of 2 × 2 × 2 mm. Two detector banks (not shown) are located to the left and right of the samples allowing for simultaneous collection of

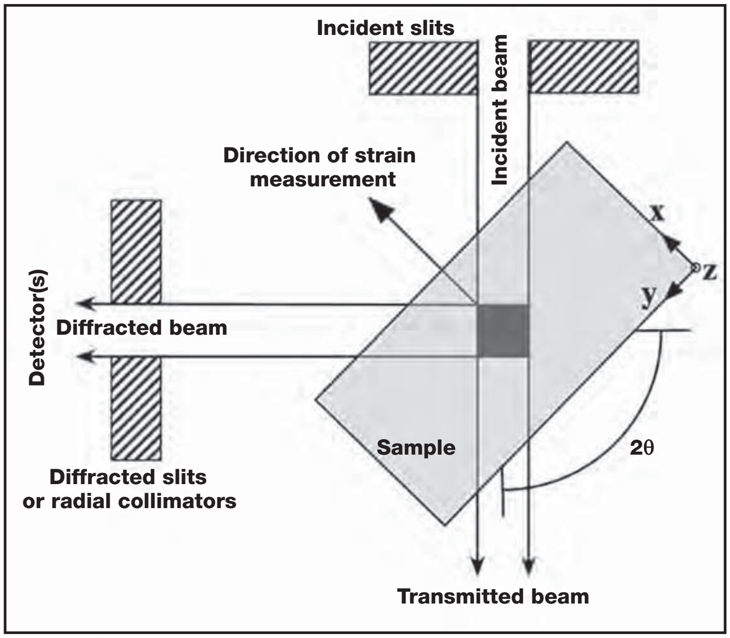

Fig. 4 — *Schematic of neutron diffraction set up viewed from above shows nominal gauge volume (dark square) as defined by the slits[7].*

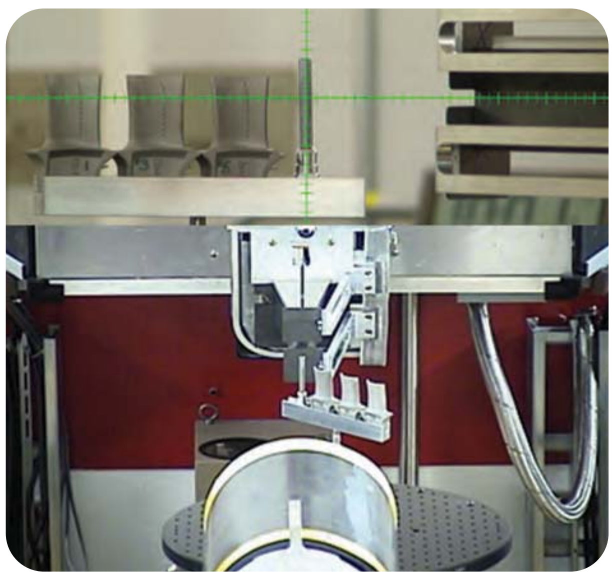

Fig. 5 — *Precision alignment of the turbine blades and powder sample (top) in the VULCAN beam line (bottom).*

two orthogonal strain components.

Time-of-flight measurement gives concurrent analysis of a series of Bragg reflections. Given the material's elastic anisotropy[9], only residual strains from the (311) reflection are reported here as the modulus for the (311) reflection is close to the bulk average. Figure 6 shows residual strains and their position within the blade for two samples in the as-built and stress relieved condition. Residual strain maps are superimposed on the representative "slice" of the blade from an auto-cad drawing. Slices A, B, C, D, and E are at 15, 12, 10, 8, and 0 mm up from the origin on the dovetail base, respectively. The arrow in slice A indicates the direction of the strains (ppm) at each location. The green and blue values originate from the as-built and stress relieved conditions, respectively. Each square and diamond in Fig. 6 is nominally 1 and 4 mm^2, respectively.

In the as-built blade, strains parallel to the long axis of the blade are more compressive on the convex surface than the concave surface, which may be related to sequencing in the DLMS build process. In general, residual strains in the as-built sample become more tensile (or less compressive) as the dovetail base is approached. At the base, residual strains return to being more compressive. After a proprietary stress relief process, residual strains become very compressive and much more uniform throughout the lower portion of the blade, suggesting that the blade will be less likely to distort during service. The combination of neutron tomography and residual strain measurement is a critical approach for examining complicated components that can be built using additive manufacturing.

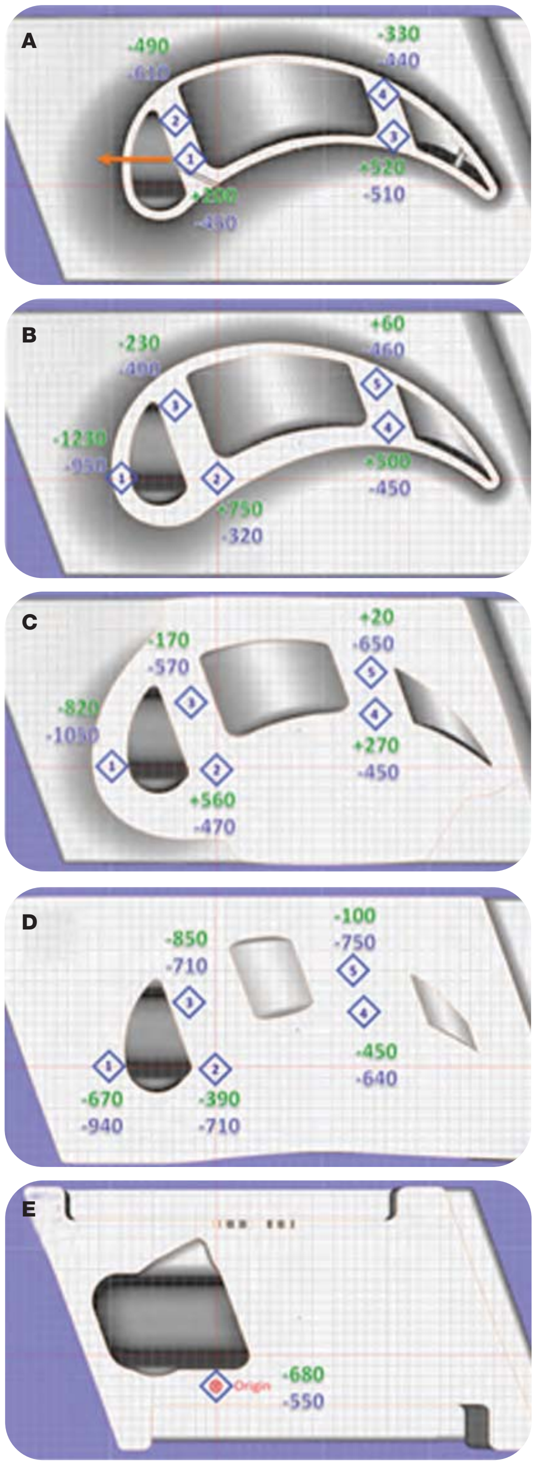

Fig. 6 — *Residual strain maps (in microstrain) of a turbine blade fabricated using additive manufacturing in the as-built (green values) and stress relieved (blue values) conditions.*

HFIR residual stress mapping

Preliminary experiments are underway at the HFIR's NRSF2 beamline (HB-2B, Fig. 7) to map stress distributions in complex parts produced by additive manufacturing. Whereas the VULCAN beamline allows for simultaneous acquisition of strains from multiple crystallographic planes, HB-2B selects a single diffraction peak as the strain sensor. Peak data are recorded for a series of sample locations and orientations to produce a residual stress map throughout the structure. Diffraction peak shifts are a measure of changes in atom-to-atom spacing, and may be related to tensile or compressive strains in the sample. Peak intensity changes may indicate preferred orientation, or changes in sample composition, and peak width may be related to crystallite size, dislocation density, and other microstructural features. The facility is being used to measure residual stress in simple prismatic shapes fabricated using various AM techniques to gain a fundamental understanding of process parameters on stress evolution. This information will be used to verify process models and stress mitigation strategies currently under development.

Summary

The use of neutrons to characterize additive manufactured components is a unique, valuable technique for imaging and measuring residual stress. Neutron and x-ray techniques can be used in a complementary fashion, providing information about the bulk and surface of a component, respectively. Neutrons are useful to characterize complex components, such as those fabricated using additive manufacturing techniques. In this article, the penetrating power of neutrons facilitated unique characterization of aircraft parts fabricated using additive manufacturing, imaging internal structures and passageways and mapping residual strains in a complex turbine blade. Future projects combining these techniques will be required to fully use the potential of additive manufacturing. ❍

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Fig. 7 — *HB-2B beamline at the High Flux Isotope Reactor.*