Hellenic Neutron Association Newsletter

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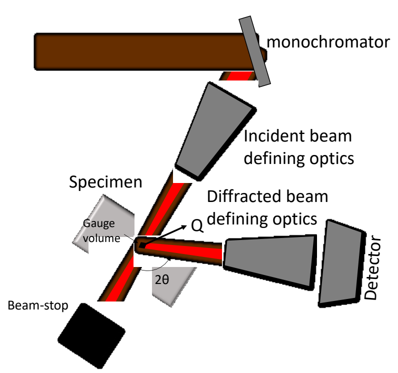
INSIDE THIS ISSUE

"Neutron diffraction, as a probe of metallic materials"



Editorial

By Konstantina Mergia (NCSR "Demokritos", Athens, Greece)

Welcome to the seventh issue of HENA newsletter!

In the current issue Dr Efthymios Polatidis, responsible for the POLDI beamline at the SINQ neutron spallation source at Paul Scherrer Institute, Switzerland, describes the use of neutron diffraction in engineering problems such as the determination of strains and stresses in engineering components and explains how the revolutionary processing technology of additive manufacturing has boosted the demand for neutron diffraction.

The latest neutron news regarding the League of advanced European Neutron Sources (LENS) and the BrightnESS2 project can be found at the end of the newsletter. Furthermore, discover how neutron science at ILL supports research on viruses such as Covid-19.

We hope you enjoy reading this newsletter and we wish you a dynamic return from the summer holidays!

How additive manufacturing of metallic parts has boosted the demand for neutron diffraction

By Dr. Efthymios Polatidis, Laboratory for Neutron Scattering and Imaging (LNS), Paul Scherrer Institute, responsible for POLDI beamline

Our society and technology have experienced three industrial revolutions: the first industrial revolution was the transition to new manufacturing processes in Europe and the United States by mechanization using water and steam power in the 19th century. The second revolution was the transition to mass production and industrialization from the late 19th century into the early 20th century. The third industrial revolution was the introduction of electronics and digitization of manufacturing from the 1970s onwards. The fourth industrial revolution, the so-called Industry 4.0, is imminent and it will fundamentally change the production methods used in the industrialized countries. A key component for Industry 4.0 is the implementation of machines that can produce components faster, more flexibly and more precisely than ever before. Additive manufacturing (AM), often called 3D printing, is a revolutionary processing technology which will play a key role towards Industry 4.0.

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AM processes of metals and alloys allow building three-dimensional parts by progressively adding thin layers of materials guided by a digital model [1,2]. By doing this, it is possible to produce near net shape, complex or customized parts directly from the design without the need for expensive machining methods. One of the most commonly used AM processes is the family of the so- called Powder Bed Fusion processes including the following methods: Direct metal laser sintering (DMLS), electron beam melting (EBM), selective heat sintering (SHS), selective laser melting (SLM) and selective laser sintering (SLS) [3,4]. In this family of AM processes, a laser beam or electron beam is used to melt and fuse the material powder together.

With the appearance of modern AM processes, building complex components from metallic powders has become highly attractive for many applications.

During building of the components layer-by-layer, the spatially varied thermal cycles that the material undergoes result in the formation of thermal expansion mismatches resulting in the development of residual stresses (RS) [5]. The RS impose high risks for the geometrical and mechanical behavior [6] and sustainability of the components built by AM processes. Therefore, a significant research effort is currently undertaken to understand the development and methods for suppressing RS, by manipulating the processing parameters or post-built treatments such as annealing [7,8], hot-isostatic pressing [9] or laser shot peening [10]. The ability to characterize RS accurately is key to this research effort.

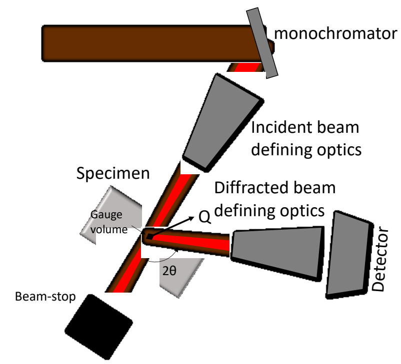

Fig. 1 Schematic illustration of a typical continuous source-based diffractometer for strain measurement.

Neutron Studies: Neutrons are electrically neutral and, in contrast with X-rays which interact dominantly with the electron shell of the atom, interact on the level of the nucleus. Most engineering materials and alloys exhibit comparatively low interaction probability with neutrons and therefore neutrons are able to probe bulk material structures. This property of neutrons is exploited in the characterization of materials and components through neutron scattering. Neutron diffraction is a powerful tool for characterizing the lattice structure and crystal lattice distortions, i.e. lattice strains, at depths which are not amendable by X-rays non-destructively as opposed to methods such as hole drilling or the contour method [11].

Precise angular position of a diffraction peak provides a lattice plane distance-d_{hkl} (averaged over all grains of the same {*hkl*}-family in the illuminated gauge volume) in accordance with the Bragg equation:

$$\lambda = 2d_{hkl}sin\theta \qquad (2)$$

where λ is the wavelength, and θ the diffraction angle. When measuring the diffraction peak position of a strained sample (d_{hkl}) and the "stress-free" reference sample ($d_{0,hkl}$), the {*hkl*}-dependent elastic lattice strain can be obtained by:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}} \qquad (3)$$

Special care is taken for the determination of the reference value of the interplanar spacing, from the "stress-free" sample, as it can greatly affect the magnitude of the calculated strain and the error in determining the $d_{0,hkl}$ value propagates and increases the uncertainty of the calculated RS magnitude [12].

In order to determine the full residual stress state first the strain tensor ε_{ij} has to be determined. This can be achieved by measuring the lattice strain along multiple directions, ideally along the three principal directions (where the shear strain components are zero). Subsequently the stress tensor is obtained using the generalized Hooke's low:

$$\sigma_{11} = \frac{E}{(1+\nu)(1-2\nu)}[(1-\nu)\varepsilon_{11} + \nu(\varepsilon_{22} + \varepsilon_{33})] \qquad (4)$$

where E is the Young's modulus and ν is the Poisson ratio. A typical example of a strain scanning instrument at reactor sources (also frequently referred to as steady state or continuous sources) is shown schematically in Fig. 1. The polychromatic neutron beam is first monochromated to a chosen wavelength using a suitable monochromator. The divergence and size of the monochromatic beam is adjusted using appropriate neutron optics in the path of the incident beam. The diffracted beam is shaped using suitable optical devices, before being collected by the neutron detector. The gauge volume over which the strain is averaged is defined by the intersection of the incident and diffracted beams (Fig. 1). The general operating principle of a neutron diffractometer at a continuous source is the same as for a conventional X-ray diffractometer. Monochromatic strain scanners are STRESS-SPEC (FRM II), HIDRA (SNS), SALSA (ILL), BT8 (NIST), HB-2B (SNS), Kowari (ANSTO), E3 (HZB) which no longer exists after the end of 2019 and many more around the globe.

At pulsed sources, or continuous sources that beam choppers are employed to produce pulsed neutrons, the neutron beam consists of a series of short pulses, each containing a spectrum of wavelengths. The energy of each neutron can be determined from its velocity (time of flight method - ToF) and thus, the entire diffraction pattern is recorded at any particular scattering angle. The strain, ε, can be measured from the time t which the neutrons scattered in a Bragg reflection *hkl* need for their flight path, and the strain is given by:

$$\varepsilon_{hkl} = \frac{t_{hkl} - t_{0,hkl}}{t_{0,hkl}} \qquad (5)$$

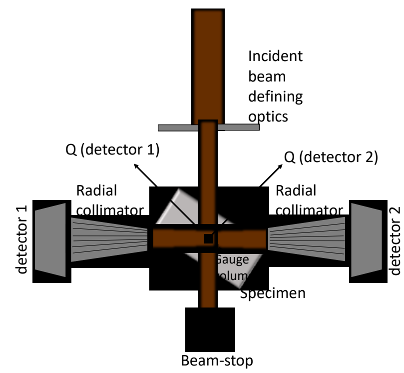

Fig. 2 Schematic illustration of a typical ToF instrument featuring two detector banks.

A schematic of a typical ToF-diffractometer used for strain measurement is shown in Fig. 2. The TOF mode is used at ENGIN-X (ISIS), SMARTS (LANSCE), VULCAN (SNS), POLDI (SINQ), TAKUMI (J-PARC), EPSILON (SKAT).

Types of neutron diffraction studies: As mentioned above, the RS impose high risks for the geometrical and mechanical integrity and sustainability of the components. Therefore, a significant research effort is currently undertaken to understand the development and relieving of RS in the as-built components, mainly by post-built treatments [13–17]. The AM processes involve numerous building parameters and afterwards, numerous post-building treatments which affect the geometrical integrity, surface quality, microstructure, mechanical behavior and many other key properties of the materials and components. Thus, a comprehensive study of the balance between building parameter/properties requires a large number of samples and multiple measurements to capture trends. The investigations of the effect of annealing temperature and time are thus usually undertaken by parametric studies, i.e. several samples are built, post-built treated at different temperatures for varying times and finally the RS are measured using neutron diffraction. Hence, typically the requested beamtime for these investigations is longer than for conventional RS characterization or other neutron diffraction studies.

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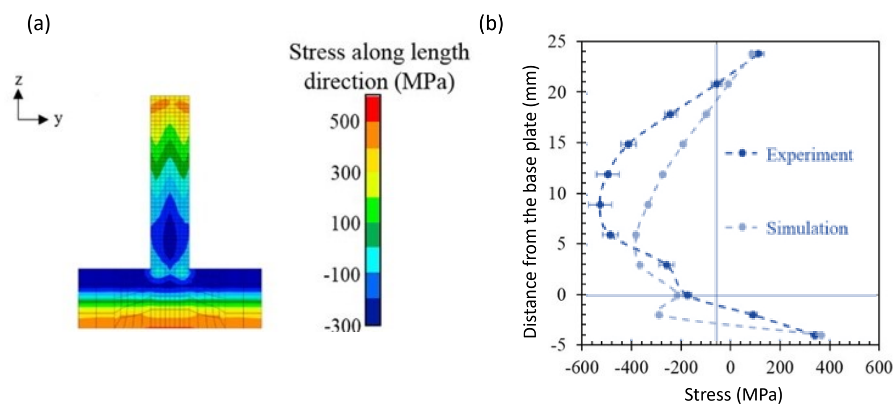

Fig. 3 (a) Schematic of finite element mesh of an Inconel 625 AM-produced wall. 2D residual stress contours (in MPa) along length direction of the Inconel 625 wall with 40 s dwell time between layers. Reproduced from [22].

An alternative route is employing numerical thermo-mechanical modeling that can predict the development of RS, however, the quality of the calculations depends critically on the accurate constitutive thermodynamic/kinetic/thermal /mechanical models that describe all the complex processes that occur during the AM processes. To this end, quantifying residual stress experimentally provides essential computational model validation [18–22]. A typical example is shown in Fig. 3 where the RS that develop in a component using Inconel 625 processed with laser-based direct energy deposition method was modelled and validated by neutron diffraction on the VULCAN instrument at SNS [22]. The agreement between experiments and calculations is excellent.

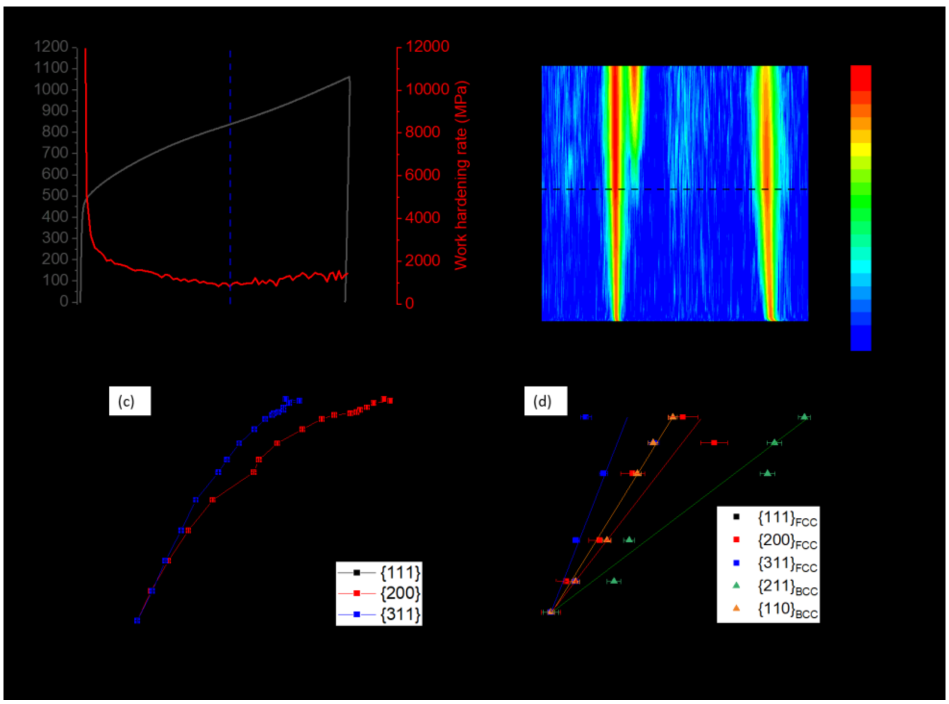

Fig. 4 (a) True stress-strain and work hardening rate showing hardening after approximately 0.23 true strain (indicated with a blue dashed line). (b) Evolution of neutron diffraction patterns showing the martensite formation (appearance of the 110_{BCC}, $10\bar{1}0_{HCP}$ and $10\bar{1}1_{HCP}$ reflections) after approximately 0.23 true strain (indicated with a black dashed line). (c) Lattice strain evolution in austenite. (d) Lattice strain evolution from 0.23 true strain until 0.42 true strain showing that martensite accumulates lattice strain at higher rate than austenite. The lines in (d) are linear fits to the experimental data for guiding the eye reproduced from [25].

Besides residual stress investigations, neutron diffraction is a well-established technique for studying microscopic strains and the evolution of the microstructure under deformation *in situ*. Samples produced with different processing parameters exhibit strong crystallographic textures, heterogeneous microstructures and grain morphologies and hence anisotropic mechanical properties. The evolution of lattice strains and diffraction peak width can provide insight on the average intergranular and intragranular strains, while peak intensity the texture/phase evolution within different grain families. The average lattice strain of a grain family represents the fraction of applied load, i.e. the macroscopic stresses shared by that grain family. In situ neutron diffraction is also useful for studying multiphase materials [23] or phase transforming materials during deformation [24,25], as it reveals the phase evolution, the load transfer between the phases and provides information on the role of each phase for the strain hardening behavior of the composite material. In a recent experimental study of a phase transforming steel produced by SLM, the hardening effect of martensite was revealed by in situ uniaxial tension during neutron diffraction, as shown in Fig. 4. It was found that upon deformation martensite appears and carries significantly more load than austenite [25]. Such advances in understanding the link between process and properties in additive manufacturing, open new opportunities for realizing advanced complex materials, with clear economic, technological and environmental benefits over conventional manufacturing methods.

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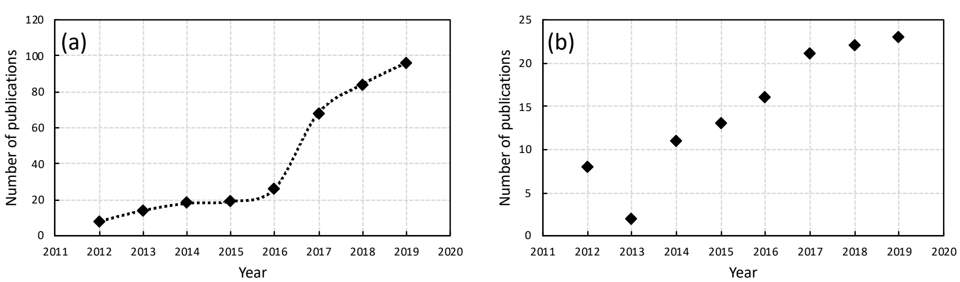

Fig. 5 (a) Number of publications including the keywords "additive" and "residual stress". (b) Number of publications including the keywords "additive", "neutron" and "diffraction" between 2006-2020. Source: Scopus®, retrieved on 26.04.2020.

Concluding remarks: Neutron diffraction is an essential tool for engineering materials science and plays a key role in the establishment and exploitation of modern processing methods, such as AM. The AM user community and the relevant industry hence, strive for accessing neutron diffractometers. This has resulted in an overall increase of beamtime demand, while since few years, on average a >30% share of the submitted beamtime proposals to engineering neutron diffractometers are associated with AM-related investigations. Fig. 5(a) and 5(b) illustrate the constantly increasing research effort in the field of neutron-related studies of residual stress and AM the past few years. Considering the world-wide instrument availability and the already high demand of such instruments, the AM-related studies hold a significant share of the requested beamtime. The user community also strives for obtaining fast and reliable measurements with good spatial resolution but also faster *in situ* or quasi *in situ* observations, which are all directly related to the instruments' performance. Such instrumental expectations push towards more brilliant sources, increase of neutron flux by more efficient neutron optics, advanced data acquisition systems for event mode streaming and more efficient detector technologies. To this end the new instrumentation for engineering studies, currently under development at the European Spallation Source ESS, i.e. BEER and ODIN instruments, will provide outstanding means for achieving break-through insights on advanced manufacturing processes. In the meantime, many of the existing neutron diffraction instruments undergo important upgrades to cope with the increasing demands of the research community.

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Neutron News

Find out about the League of advanced European Neutron Sources (LENS) League and read the associated news, neutron related webinars and scientific highlights in the link https://www.lens-initiative.org/

Read how neutron science at ILL supports research on viruses such as Covid-19.

Read about the recent advances of the BrightnESS2 project in the link https://brightness.esss.se/news-and-events