PROFILOMETRY-BASED INDENTATION PLASTOMETRY BRINGS SPEED AND ACCURACY TO METALLURGICAL R&D

This unique testing method offers economic benefits, sustainability advantages, and the potential to slash the time required to develop new alloys and processing recipes.

Bryer C. Sousa and Danielle L. Cote**
Worcester Polytechnic Institute, Massachusetts
Matthew A. Priddy
Mississippi State University
*Victor K. Champagne, Jr.**
U.S. Army Research Laboratory, Maryland

Profilometry-based indentation plastometry (PIP) was developed and commercialized by Plastometrex in the U.K. This unique testing method offers the materials science, engineering, and processing community a paradigm shift in the mechanical characterization of metal systems. Rooted in indentation, PIP testing gives metallurgical and mechanical engineers a simple way to determine nominal stress-strain curves up to the ultimate tensile strength (the point at which the onset of plastic instability arises), as well as true stress-strain curves, Voce plasticity parameters, yield strength, and simulated Brinell hardness values in less than five minutes.

Another notable feature is that PIP testing only requires a sample thickness of roughly 2 mm, and length and width dimensions of just 6 mm. This can lead to significant cost savings compared with sample requirements for uniaxial tensile testing, specimen machining, and component manufacturing. For example, assuming a rectangular specimen geometry, the volume of material required for PIP testing per stress-strain curve is 72 mm^3, whereas an ASTM E8 sub-size specimen (prior to machining to test coupon geometry) requires a volume of 2100 mm^3 per stress-strain curve. The result is a reduction in material costs of approximately 97% when using PIP testing and plasticity analysis.

PIP THEORY AND PRACTICE

PIP is performed using an indentation plastometer (Fig. 1). As noted by Tang et al., PIP-obtained tensile stress-strain curves are derived by loading a hard spherical tip into a given specimen until reaching a known force[1]. This is followed by measuring the resultant indent profile and performing an iterative finite element model (FEM) simulation of the same test until best-fit plasticity parameters are achieved. More details regarding the process of obtaining tensile stress-strain curves via PIP analysis will be discussed shortly; the true stress-strain relationship (metallic plasticity behavior) and deformation response of the material are formulated via the Voce plasticity model, as far as an analytical framework for PIP testing of strain hardening material systems is concerned[2].

Numerous constitutive plasticity laws and analytical expressions are found in the literature, such as those presented by Hollomon, Swift, Ludwik, Harley and Srinivasan, Ludwigson, and Baragar[3]. However, the implementation of a Voce plasticity framework within PIP testing centers on the observation that the Voce model was capable of characterizing the plasticity response of metallic material systems with true strain hardening rates that approach zero. Further, the Voce model was also the most adept plasticity model for effectively capturing accurate, true stress-strain behaviors across a range of alloys and metals.

In contrast with the Ludwik-Hollomon plasticity model, which is expressed as:

$$\sigma = \sigma_y + K\varepsilon^n$$

wherein σ represents the von Mises applied stress, σ_y represents the von Mises yield stress, ε represents the von Mises plastic strain, *K* represents the strain

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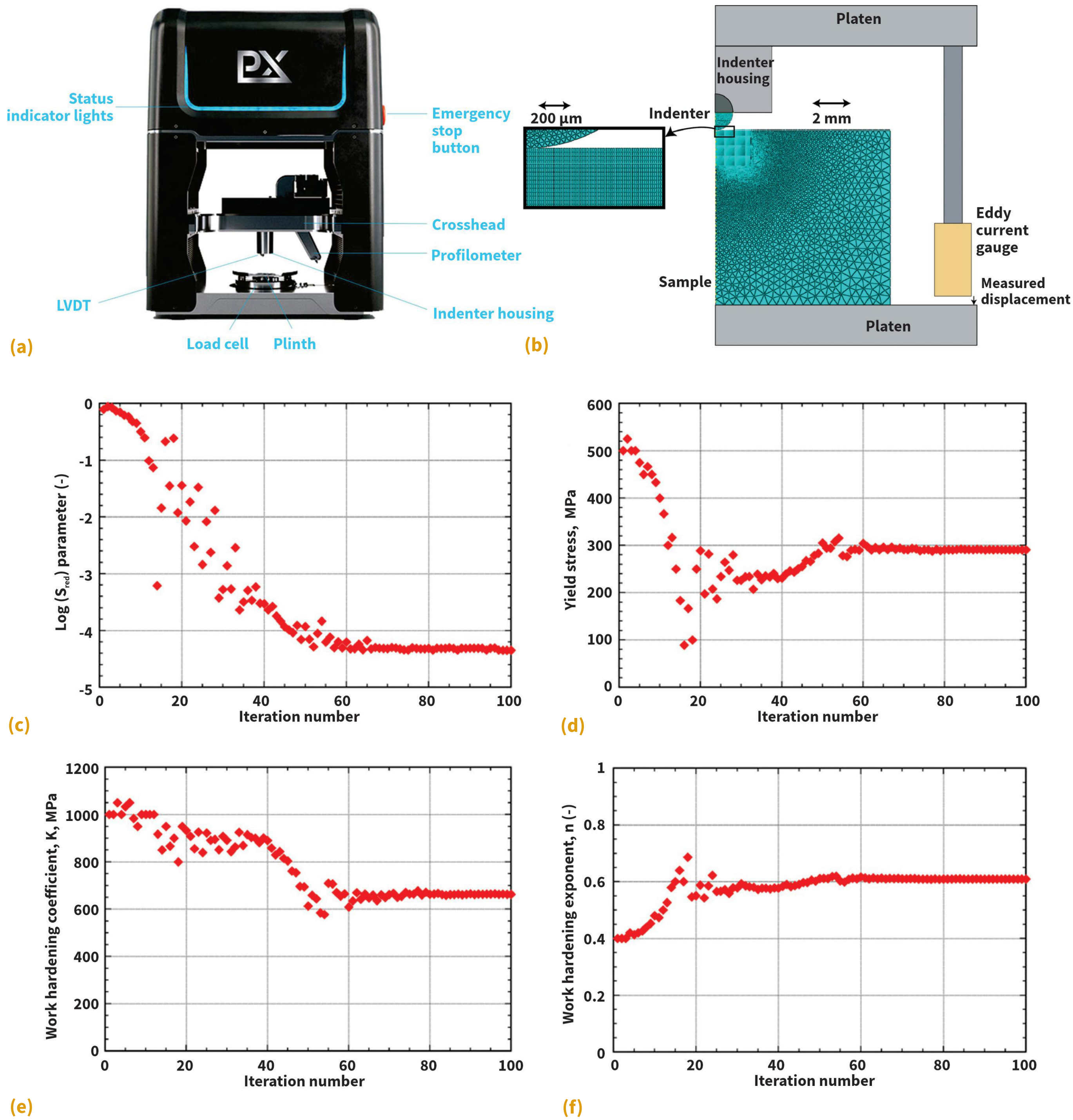

Fig. 1 — (a) Indentation plastometer from Plastometrex; (b) FEM-based schematic of a plastometer for PIP testing; and (c, d, e, and f), the convergence process underpinning PIP data analysis[2].

hardening coefficient, and n represents the strain hardening exponent, the Voce plasticity model is expressed as:

$$\sigma = \sigma_s - (\sigma_s - \sigma_y) \exp\left(\frac{-\varepsilon}{\varepsilon_o}\right)$$

wherein σ, σ_y, and ε are the same variables found in the Ludwik-Hollomon plasticity model expression, σ_s is the saturation stress, and ε_o is the characteristic strain. Reference 3 is a valuable resource for more information related to PIP testing.

USING PIP IN MATERIALS PROCESSING

The streamlined PIP testing method could benefit a wide range of applications within the advanced materials and processing community. For example, consider the case of capturing plasticity parameters and intrinsic mechanical properties as a function of thermomechanical processing times and temperatures along with standard aging curves, which traditionally rely on indentation hardness or tensile test data as a function of processing parameters. In contrast, PIP testing can more easily identify optimal processing parameters in a high-throughput manner.

As such, heat treatment optimization and tuning for application-specific performance requirements are well suited for PIP testing and analysis integration. This is especially relevant for the aluminum, steel, nickel, titanium, cobalt, and copper-centric domains of expertise. Accordingly, the Cote Research Laboratory at Worcester Polytechnic Institute (WPI) is actively working with Solvus Global, also located in Worcester, Mass., the U.S. Army Research Laboratory, the University of Massachusetts Amherst, the Metals Processing Institute, Florida International University, Mississippi State University, and others to utilize PIP in this way further. For example, the thermal post-processing optimization of wire arc additive manufacturing (WAAM)-based maraging 250 steel systems is underway. Beyond heat treatment optimization, advanced processing parameters are being evaluated for cold spray additive manufacturing (CSAM), WAAM, photopolymer additive manufacturing (PAM), directed energy deposition (DED), and additional materials processing methods.

CASE STUDY 1: COMPARISON OF PROCESSING TECHNIQUES

To demonstrate the utility of PIP testing within the realm of processing method selection, Al 6xxx specimens were produced via high-pressure nitrogen CSAM, a hybrid combination of shot peening and high-pressure nitrogen CSAM, arc melting, and extrusion. The CSAM-based Al 6xxx consolidations were processed using a 0071 polybenzimidazole nozzle, a cold spray system from VRC Metal Systems, Box Elder, S.D., an inertly gas-atomized Al 6061 feedstock powder, and a wrought Al 6061-T651 build plate. The arc melted specimen was produced using wrought Al 6061-T651 and an electric arc as a thermal energy source.

As for the hybrid combination of shot peening and high-pressure nitrogen CSAM consolidations, the same processing parameters, feedstock, hardware, and system were used; however, shot peening was introduced at constant intervals of deposited layers. Further, while a roughness of approximately 3 µm or less is best for PIP testing, specimens were compression mounted using phenolic mounting material and a SimpliMet 4000 system from Buehler, Lake Bluff, Ill. This was followed by grinding and polishing for metallographic preparation using a Buehler EcoMet 300 automatic polisher until a mirror finish was achieved using a 0.02 µm colloidal silica suspension. After that, PIP testing was performed per prescribed protocols, execution of the tests via CORSICA 2.0, and analysis via SEMPID (software for extracting material properties from indentation data). Figure 2 shows testing results and analysis.

In addition to the nominal or engineering stress-strain curves obtained and presented for the arc melted, hybrid CSAM and shot-peened, high-pressure CSAM deposited, and extruded Al 6xxx specimens, one can identify that the processing technique achieves the desired

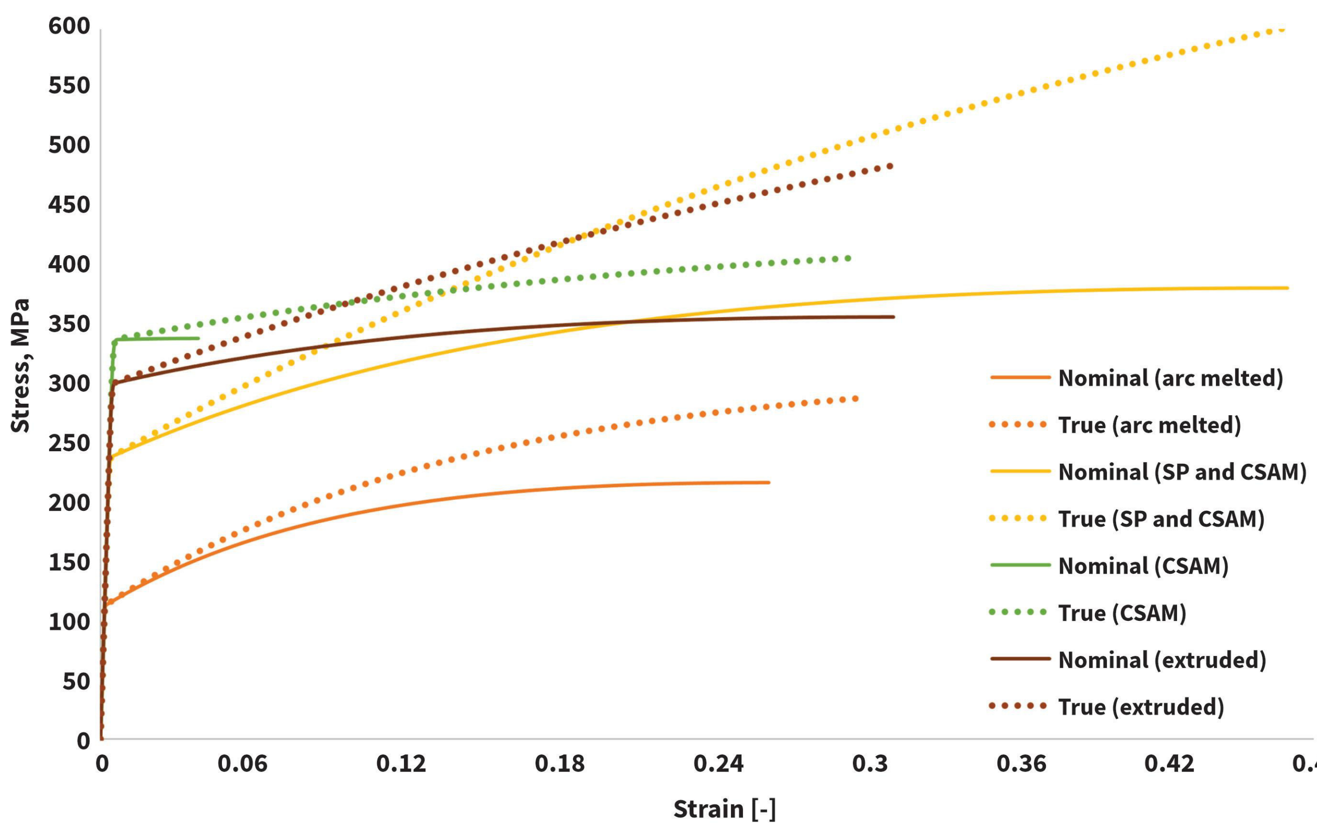

Fig. 2 — Nominal and true stress-strain curves for the four uniquely processed Al 6xxx series studied here.

strength-to-ultimate tensile strain ratios for a given application. The arc melted material was found to have the lowest yield strength (113 MPa) and lowest ultimate tensile strength (217 MPa). Regarding yield strengths (excluding the arc melted specimen discussed above), the hybrid CSAM and shot-peened, extruded, and CSAM processed Al 6xxx specimens were found to be 238, 300, and 338 MPa, respectively. At the same time, the ultimate tensile strengths (excluding the arc melted specimen) for the hybrid CSAM and shot-peened, extruded, and CSAM processed Al 6xxx specimens were found to be 381, 356, and 339 MPa, respectively.

By coupling these insights regarding strength with implications for ductility from the nominal stress-strain curves obtained via PIP testing and analysis, hybrid CSAM and shot-peened processed Al 6061 resulted in the most pronounced balance between strength and ultimate tensile strain, i.e., the strain at which the onset of plastic instability initializes, followed by necking until critical failure occurs. Interestingly, one can demonstrate the value of using the Voce plasticity constitutive law parameters obtained from PIP testing to define the mechanical characteristics within FEA and affiliated models to obtain insights surrounding specimen-specific deformation behaviors. For example, SEMPID contains a tool for PIP users that provides operators with the ability to model Brinell indentation as a function of the Voce plasticity parameters garnered from PIP analysis (Fig. 3).

Specifically, this built-in feature within SEMPID models a Brinell indentation test wherein the simulated Brinell indenter tip radius is 5 mm and the max indentation load applied is 3000 kg. Thus, Fig. 3 captures the final effective plastic strain fields and von Mises stress fields for the hybrid CSAM and shot-peened, extruded, and CSAM processed Al 6xxx specimens at the maximal external load applied. Concurrently, those interested can also obtain similar plots at various stages of external loading and plot displacement fields and Brinell residual indent profiles. In any case, for the extruded Al 6xxx, CSAM processed, and hybridized CSAM and intermittently shot-peened specimens, Brinell test simulations recorded Brinell hardness values and indent diameters of 123.9 kg/mm^2 and 5.333 mm, 121.1 kg/mm^2 and 5.389 mm, and 113.3 kg/mm^2 and 5.556 mm, respectively. SEMPID software also enables users to model spherical indentation and uniaxial tensile tests using the plasticity parameters measured and the axisymmetric tensile coupon geometries and measured ductility. Note that the user must define both of these latter variables prior to performing computational analysis.

CASE STUDY 2: WAAM APPLICATIONS

This case study focuses on wire arc additive manufacturing (WAAM) and highlights PIP within the context of post-processing influence on strength. The research involved applying PIP testing to a WAAM-processed maraging 250 steel material system. More specifically, the as-printed and stress relieved WAAM-processed maraging 250 steel systems were produced using constant processing parameters and build plate compositions. Stress relief thermal post-processing was first performed on the WAAM-processed consolidations attached to the build plate. Accordingly, PIP testing enabled insights into the stress-strain behavioral evolution induced by thermal post-processing compared to the as-processed counterpart. The two WAAM-processed specimens are shown in Fig. 4 before metallographic preparation and PIP testing. Nominal and true stress-strain data for both

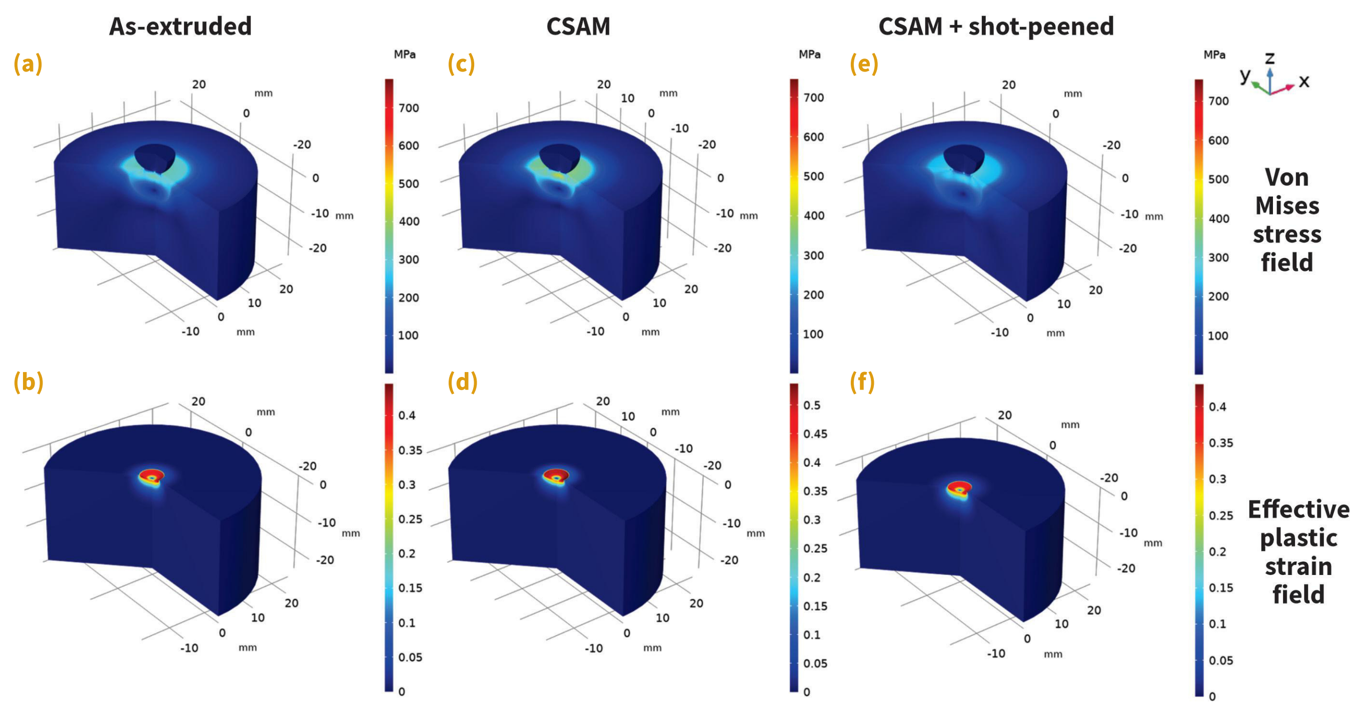

Fig. 3 — PIP and SEMPID-enabled FEA of simulated Brinell indentation testing of three uniquely processed Al 6xxx materials.

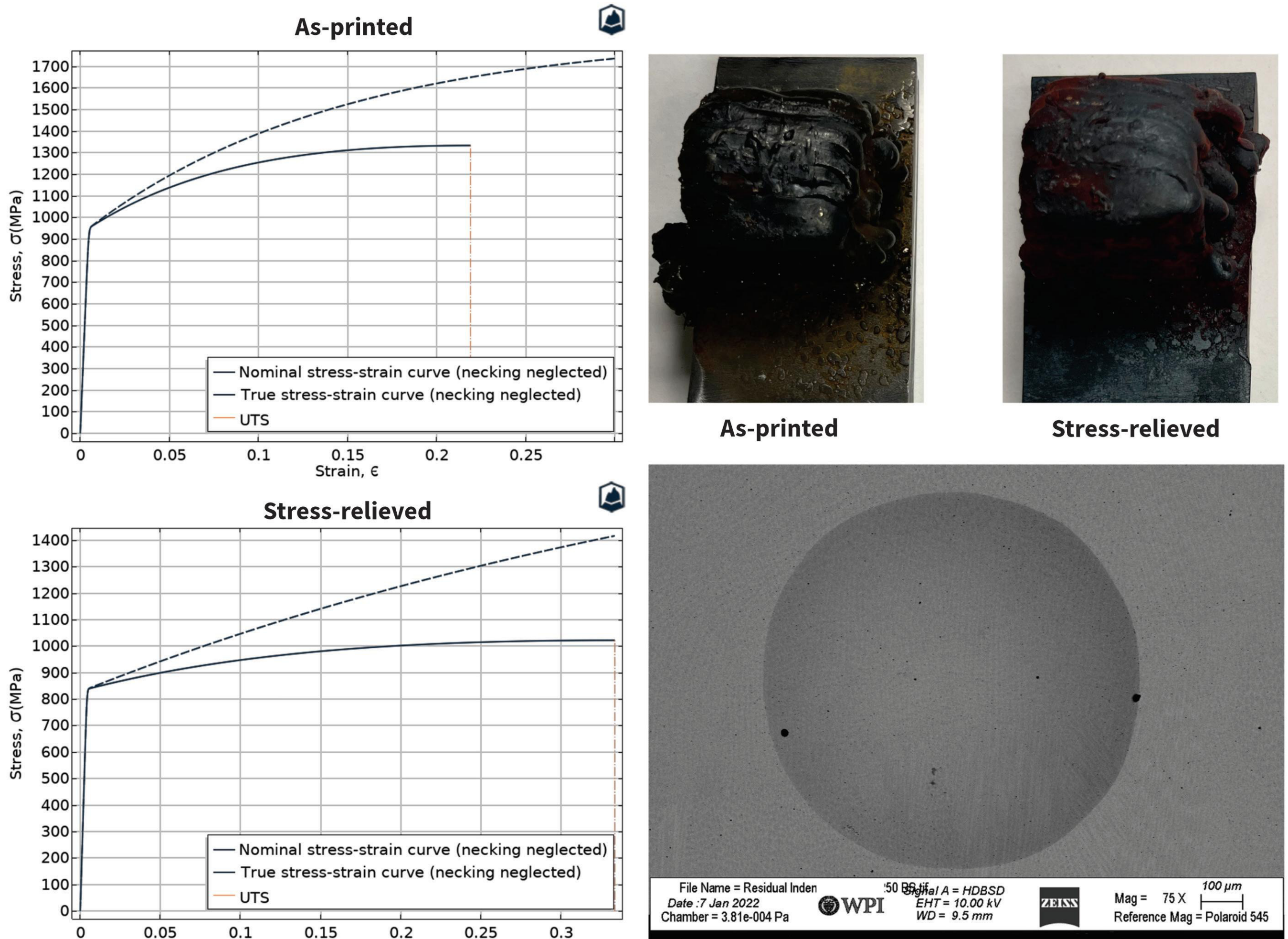

Fig. 4 — PIP analysis of as-printed and stress-relieved WAAM-processed maraging 250 steel systems.

conditions are presented alongside a scanning electron micrograph of a residual PIP indent applied to a WAAM-processed maraging 250 steel system.

PIP testing and analysis found the yield strength, ultimate tensile strength, and characteristic strain for both the as-printed and heat treated conditions to be 950 and 838 MPa, 1333 and 1022 MPa, and 13% and 50%, respectively. Testing was conducted parallel to the build direction and equidistant from the deposit-substrate interface and specimen surface. Soon, the degree of information obtained via PIP testing and analysis of the WAAM-processed steels discussed here will be enhanced. PIP line scans are currently being measured for region-specific property insights; multiple orientations relative to the build, traverse, and step directions are being characterized; substrate-free WAAM-processed and thermally post-processed specimens are being characterized to explore the relationship between residual stress states and resultant PIP behavior.

CONSTRAINTS AND LIMITATIONS

Like all materials characterization methods, PIP has its limitations. For example, PIP testing should not be performed within 3 mm of a specimen's given edge. In addition, one must avoid near-edge measurements to ensure that the presence of both free edges and the edge of the specimen in contact with the mounting matrix material will not influence the plastic deformation field formed during testing. This could reduce PIP test accuracy due to the potential for deviations from radial symmetry, which would violate the underlying principles within the indentation models.

For high-fidelity PIP testing and analysis, one must also consider specimen curvature and surface roughness variation boundaries. Currently, the maximal radius of curvature is 1 m, and the maximal surface roughness is 10 µm; however, surface roughness of 3 µm or less, or surfaces that have been polished using P1200-P2400 grit polishing paper, is ideal for PIP applications. To date, materials that can be tested via PIP include pure and alloyed aluminum, steel, titanium, nickel, copper, and cobalt systems. Note that materials with yield strengths well above 2000-3000 MPa are also currently out of range.

RECENT PROGRESS

PIP testing and analysis are steadily gaining wider adoption and further integration within various sectors of the metallurgical and materials engineering communities. Recent advancements have resulted in the application of PIP testing and analysis to the following metallic materials:

- Brazed and nonbrazed Hardox steels used for mining applications[4]
- Forged aluminum systems[5]
- Fusion welds[6]
- Residual stress quantification and effects upon PIP data[7,8]
- Quantification of creep characteristics[9]

With the extensive PIP testing and analysis capabilities, the advanced materials and processing community is well positioned to benefit from this novel characterization method. **~AM&P**

For more information: Bryer C. Sousa, doctoral candidate, Department of Mechanical and Materials Engineering, Worcester Polytechnic Institute, 100 Institute Rd., Worcester, MA 01609, bcsousa@wpi.edu.

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