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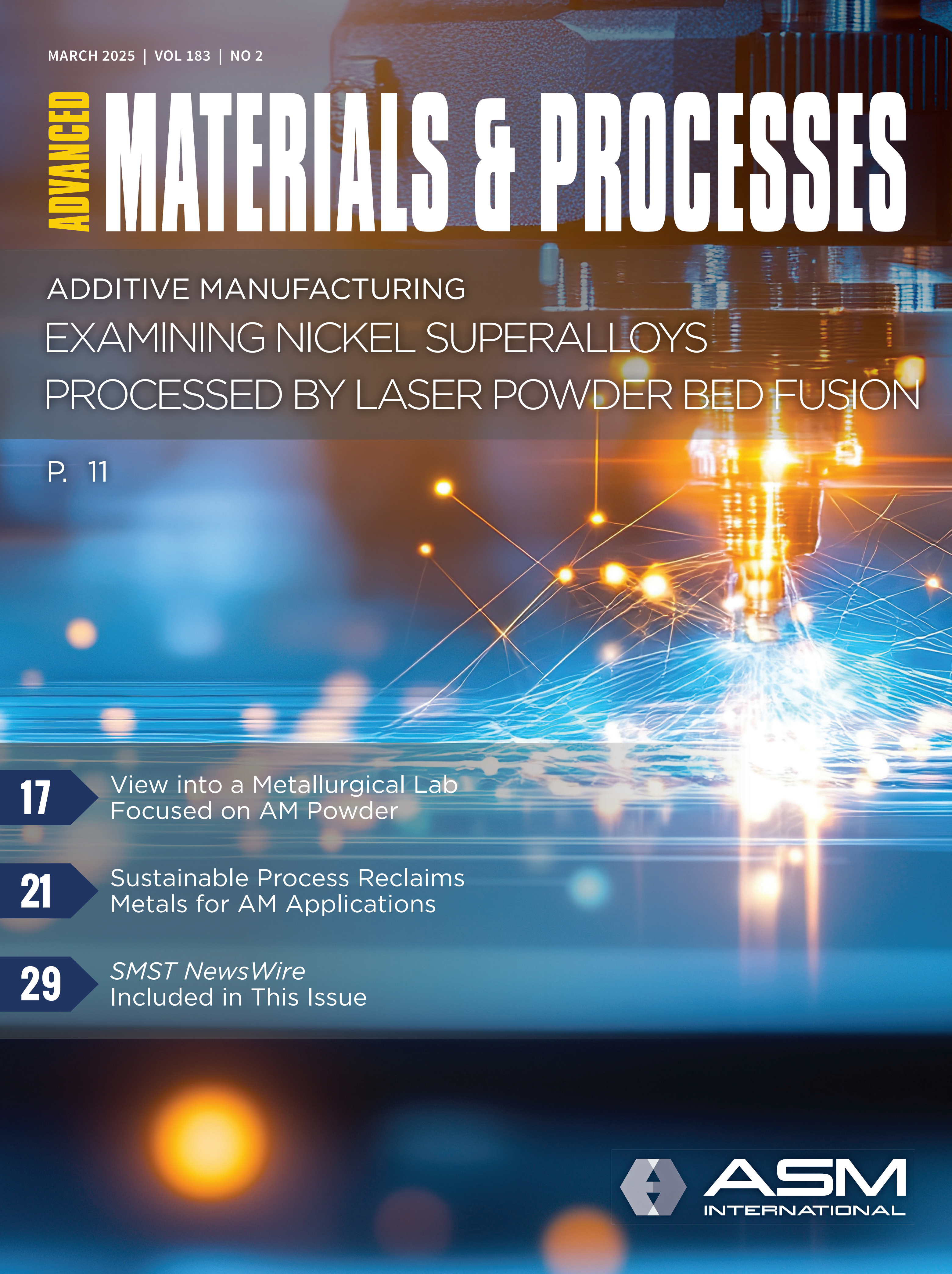
ADVANCED MATERIALS & PROCESSES



ADDITIVE MANUFACTURING
EXAMINING NICKEL SUPERALLOYS PROCESSED BY LASER POWDER BED FUSION

P. 11

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DO SHAPE MEMORY ALLOYS HAVE STANDARDS?

A growing collection of standard material specs and test methods has been established to assess and control quality in shape memory alloys for medical devices and actuator applications.

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In the early 1990s the use of superelastic shape memory alloy (SMA) for medical devices was on the verge of a multi-billion-dollar industry with no standard specifications and test methods available[1]. This motivated the development of the first set of standard terminology, specifications and test methods for NiTi based SMA[2,3]. Here, the SMA community focused on characterizing and optimizing processes for a single binary NiTi alloy system with 54.5 to 57.0 wt% nickel and niche applications[2]. In the early 2000s the first set of ASTM were published, which included F2004[4], F2005[5], F2063[6], and F208[7] for binary NiTi for medical device material and test methods. At the time, it was already well known that composition, processing, metallurgical structure, and heat treatment had a significant influence on the thermal and mechanical properties and fatigue life of SMAs in the end-product[8,9]. With this in consideration, F2063 was created to specify requirements and test methods for verifying chemical composition, transformation temperatures, metallurgical structure (including grain-size, porosity, and nonmetallic inclusions), and mechanical properties of wrought binary NiTi. Non-SMA specific test methods are described in F2063 for analyzing chemical composition[10], grain-size[11], nonmetallic inclusions[12], and mechanical properties[13]. ASTM test methods

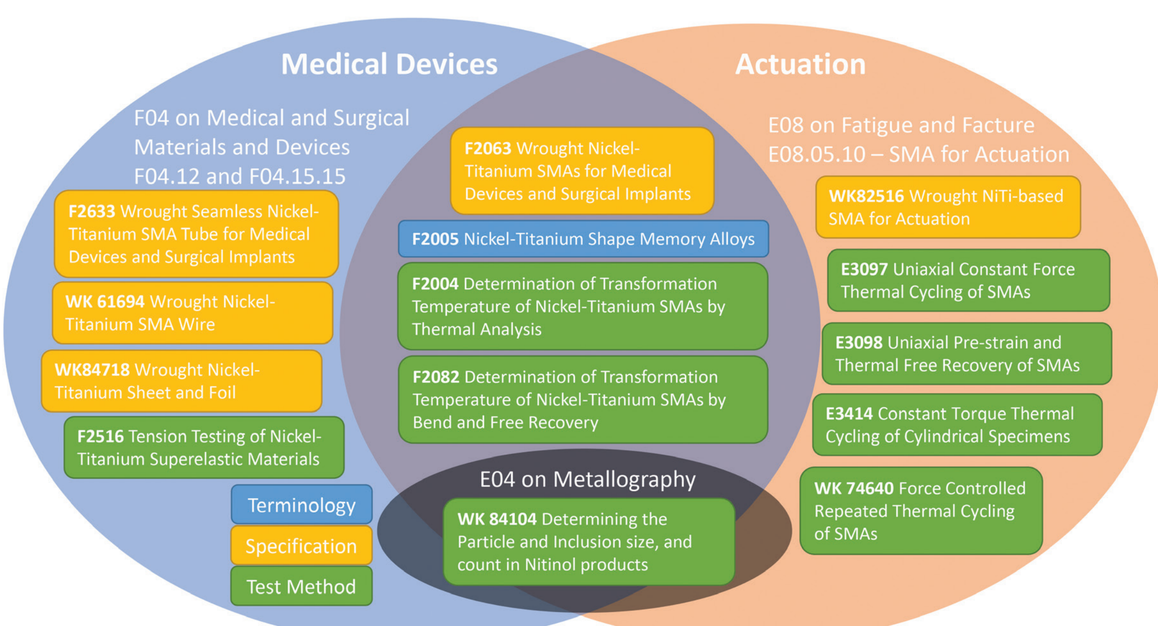

Fig. 1 — Ecosystem of ASTM shape memory alloy standards for medical devices and actuation.

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F2004 and F2082 were created to determine transformation temperatures by differential scanning calorimetry (DSC) and bend and free recovery, respectively, and ASTM F2005 defines standard terminology for NiTi-based SMAs. These specifications and test methods are currently maintained by ASTM task groups F04.12—Metallurgical Materials and F04.15.15—Nitinol Test Methods.

Over the past two decades, nonmedical industries, including automotive, aerospace, and oil and gas, have seen successful commercial application of SMA actuation without standards[14]. In many cases, these applications relied on internal qualification procedures or, in some cases, the standards for superelastic SMAs developed primarily for medical applications. Figure 1 shows the shared usage of the core medical SMA standards as indicated by the overlap in the medical and actuation communities. The nonmedical industries and actuator applications could experience more widespread use of SMA applications with optimized manufacturing processes and quality assurance guided by standards developed specifically for actuation. Around 2020, with an initial focus on certification of aerospace SMA actuator application[15] the first set of test methods for SMA actuation were published[16]. This included E3097—Standard Test Method for Uniaxial Constant Force Thermal Cycling (UCFTC)[17], E3098—Standard Test Method for Uniaxial Pre-strain and Thermal Free Recovery (UPFR)[18], and E3414—Standard Test Method for Constant Torque Thermal Cycling[19]. These test methods are currently maintained by task group ASTM task group E08.05.10—SMA for actuation with a scope that includes specifications and test methods for alloys that undergo a thermally induced phase transformation. The overview provided here is focused primarily on ASTM SMA standards, however the VDI-German Guidelines[20] and JIS-Japanese Industrial Standards[21] also develop and maintain standards and best practices for both SMA medical devices and actuation.

DEVELOPMENT ACTIVITIES

Additional standards and revisions are needed. There are ongoing standards development activities, several of which are described here. Contact information for each ASTM work item (WK) and elastocaloric activities can be found in Table 1.

WK 84104 Determining the Particle and Inclusion Size, and Count in Nitinol Products. In the context of multiple failed interlaboratory studies in ASTM committee F04.12 on Metallurgical Materials to determine a precision and bias statement for F2063 inclusion content, it was realized that existing test methods for nonmetallic inclusions were inadequate for NiTi-based SMAs. A main application in industry is writing Nitinol material specifications and the testing thereof between suppliers and purchasers. Therefore, a current effort (ASTM WK 84104) is developing a new Standard Test Method for Determining the Porosity and Inclusion Content of Nickel Titanium Shape-Memory Alloys Using Image Analysis. The new test method, under the responsibility of ASTM committee E04.14 on Quantitative Metallography, should be applicable across industries and materials (not limited to medical or pseudoelastic). It will cover sample preparation techniques, imaging procedures, and digital image analysis. The initial focus of the new ASTM test method is on testing hot-worked product, per F2063, using scanning electron microscopy (SEM) in backscatter mode and optical microscopy. A one-lab repeatability study will be conducted upon initial publication to satisfy ASTM Form and Style Manual requirements for a new standard test method. A full international interlaboratory study (ILS) on the test method will be conducted in the future.

The working group plans to expand the scope of the standard in future revisions to also cover semi-finished products (e.g., bar, wire, and tube), as well as final devices or components. It is also envisioned to add another section later, elaborating on the application of ASTM E2283[22] on extreme value analysis of steels. It will advise on how to use this technique for a better understanding of the fatigue performance of Nitinol materials (e.g., mean free path between inclusions, nearest neighbor concept, volume at risk in an application). A first round of balloting was completed in January 2025, with courtesy ballots to committees E08 on Fatigue and Fracture and F04.12 Metallurgical Materials and F04.15 Test Methods, both for Medical Devices and Surgical Materials and Devices. These courtesy ballots were intended to reach the many Nitinol users that reside in these two major ASTM committees, allowing the gathering of broad feedback from the global Nitinol community on the new draft test method.

WK 90888 Revision of F2063, Wrought NiTi-based SMA for Medical Devices and Surgical Implants. For decades, this specification has been the cornerstone of Nitinol material standards and testing methods, supporting its commercialization in medical applications. The latest initiative involves forming task group WK 90888 to revise the specification, focusing on material grading based on micro-cleanliness and expanding considerations beyond binary Nitinol. A key aspect of this revision is refining terminology to ensure consistency and precision, particularly in product classifications. One critical update is distinguishing between “hollow” and “tube.” While “hollow” is a form of mill product, the current F2063-18 specification incorrectly classifies “tube” as such, whereas it should be categorized as a semi-finished product. Additionally, the terms “billet” and “coil” are introduced to provide a more comprehensive product classification system, addressing gaps in the current version.

TABLE 1 – RESOURCES FOR MORE INFORMATION ON SMA STANDARDS DEVELOPMENT

ASTM work items (WK) and activities	Contact	Contact email
WK 84104	Dr. Matthias Frotscher	matthias.frotscher@biotronik.com
WK 90888	Dr. Weimin Yin	wyin@resonetics.com
WK 61694, WK 84718	Dr. Maximilien E. Launey	maximilien.launey@g-rau.com
WK 82516	Dean Pick	dean.pick@kiniticsautomation.com
WK 74640	Dr. Doug Nicholson	douglas.e.nicholson@boeing.com
Elastocaloric standards	Prof. Dr.-Ing. Paul Motzki	paul.motzki@zema.de

After extensive discussions, the task group concluded that F2063 should remain exclusive to binary Nitinol, given its predominant use in medical applications. Currently complex alloys and PVD products lack dedicated specifications. While referencing F2063 for these materials is a common practice, it is technically inaccurate. The proposed solution is to develop distinct standards for these specialized materials while keeping F2063 streamlined for binary Nitinol medical industry users. Another major focus is defining material grades based on micro-cleanliness. The existing metrics—maximum inclusion size and area fraction—are overly simplistic. More robust methods, such as inclusion density and extreme value analysis for predicting maximum inclusion dimensions, may provide better classification criteria. Since micro-cleanliness evolves through thermo-mechanical processing and directly affects medical components under varying design and stress conditions, further research is needed. Establishing a strong predictive correlation between new metrics and cyclic fatigue life is essential before integrating them into ASTM standards for semi-finished Nitinol products (wire, tube, sheet, etc.).

WK 61694 Specification for Wrought Nickel-Titanium Wire for SMA Medical Devices and Surgical Implants. No standard specification defining requirements for nickel-titanium shape memory alloy wire for use in medical devices and surgical implants exists. This standard specification will be used by medical device companies, metal wire producers and government agencies to ensure consistent product and assist with quality control. This specification covers the requirements for wrought nickel-titanium shape memory alloy wire, nominally 54.5 to 57.0 mass/mass (weight) % nickel, used for the manufacture of medical devices and surgical implants. Material shall conform to the applicable requirements of Specification F2063. This specification addresses those product variables that differentiate drawn medical grade wire from the raw material and mill product forms covered in Specification F2063.

WK 84718 Specification for Wrought Nickel-Titanium SMA Sheet and Foil for Medical Devices and Surgical Implants. There is no standard specification to address the requirements specifically for nickel-titanium shape memory alloy flat rolled plate, sheet, and foil. This proposed new standard specification will be used by medical device companies, metal producers, and government agencies to ensure consistent product and assist with quality control. This specification covers the requirements for wrought nickel-titanium shape memory alloy flat rolled products including plate, sheet, and foil, nominally 54.5 to 57.0 mass/mass (weight) % nickel, used for the manufacture of medical devices and surgical implants. Material shall conform to the applicable requirements of Specification F2063. This specification addresses those product variables that differentiate flat rolled plate, sheet and foil from the raw material and mill product forms covered in Specification F2063.

WK 82516 Wrought NiTi-based SMA for Actuation. While there exists a broad range of potential applications for SMA actuators, their transition to production is hindered by a lack of standardized material specifications. To address this need, a new specification that evaluates and controls the wrought NiTi-based material for actuation is being developed under ASTM work item WK 82516. The draft specification considers several grades of material including ternary alloys and high-transition temperature Ti-rich material. More generally, a grading system has been established to categorize alloys by transition temperature and micro-cleanliness, two key considerations when selecting material for use in actuation applications. While wrought material itself is not suitable for direct use in actuation applications, follow-on specifications covering processed and finished forms are anticipated.

It is recognized that, due to chemical composition overlap between the new specification and the specification for Wrought NiTi SMAs for Medical Devices and Surgical Implants (F2063), there is potential for confusion within industry. While the main differentiator can be found within the specifications' respective scope statements, it is helpful to recognize the fundamental differences in practical application. Use cases in the medical industry predominantly utilize NiTi material for its superelasticity, defined as the material's elastic response to an applied stress. This is in stark contrast to the thermally induced shape memory effect utilized in actuation application. With so many potential branch paths and final forms it will be exciting to see how future specifications maximize the impact of NiTi-based material in a wide range of applications.

WK 74640 Constant Force Repeated Thermal Cycling (CFRTC) of SMAs. This test method is being developed to address one of the most common types of SMA thermo-mechanical responses relevant to SMA actuation. That is, applying a constant load to the SMA and repeatedly thermally cycling it through the phase transformation. During CFRTC, the SMA materials actuation strain and transformation temperatures will typically evolve. This behavior is generally undesirable for actuator applications and is often attributed to "fatigue" in SMA. This is not to be confused with conventional material fatigue and failure from fracture under a fluctuating load, although in many cases it could be similar. The objective of this test method is to assess the ability of SMA material and components to meet life expectancy requirements in an actuator application. Life expectancy requirements may include structural and/or functional fatigue limits. It is well known that chemistry, processing, metallurgical structure, and cycling parameters, including stress, lower/upper cycle temperature, and heating/cooling rates will impact CFRTC structural and functional fatigue response in SMA. This was recently highlighted in an *SMST NewsWire* for NiTiHf actuator SMA[23]. This test method, in conjunction with the material specification (WK 82516) under development, seeks to quantify, assess, and standardize these parameters for SMA actuator material.

FUTURE WORK

The newest application field for SMA is the emerging technology field of elastocalorics. The elastocaloric effect is based on the release and absorption of latent heats during mechanical loading and unloading of superelastic SMA materials. This effect is utilized in next generation heating, ventilation, and air conditioning (HVAC) systems and heat pumps, using the SMA as a solid-state refrigerant in a thermodynamic cycle. Elastocalorics is drawing increasing attention as the solid-state refrigerant offers a climate-friendly cooling alternative and latest SMA material developments suggest material COPs of 30 and higher (COP is the coefficient of performance—latent heats released/absorbed divided by mechanical work necessary). Transferring these high material COPs into commercial AC devices will cut down energy consumption for cooling (and heating) by factors.

This new way of using SMA materials comes with completely new requirements for these materials and new testing methods are required. Relevant material parameters include high internal latent heats, low mechanical hysteresis, and low transformation stress and strain, to name a few. While typical latent heat analysis has been conducted via DSC measurements, latent heats for elastocalorics need to be quantified while the material undergoes mechanical loading and unloading. Therefore, a new material testing regime has been proposed covering the complete thermo-mechano-caloric material characterization[24]. While there is intense research in the field of finding new SMA alloy systems for elastocalorics, standards in testing and characterization are needed for general comparisons and material classifications.

SMA standards are in the infancy of their development and mostly limited to niche uses. Much can be learned from alloy systems with well-established industries. The steel, aluminum, and titanium alloy industries have thousands of standard material specifications and test methods. Where practical, these standards can be divided into subcategories based on their product form, intended application, service requirement, or other similar criteria[25]. Future work may include the development of standard specifications and test methods that address forming and post-processing into semi-finished and finished components, fracture toughness, application or component specific requirements, and other areas identified by the committees. Today, as a community, we should collectively look at the suite of standards available for phase change materials and develop best practices for their use. We should also identify areas for improvement and additional standards needed based on both current and future needs.**~SMST**

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