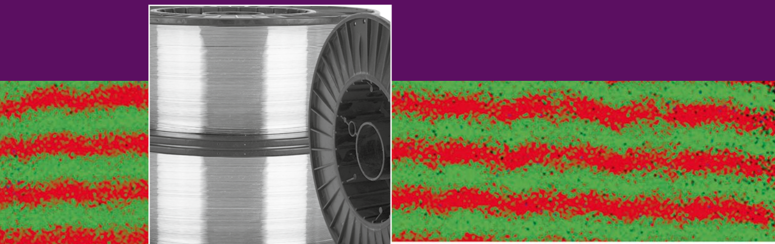
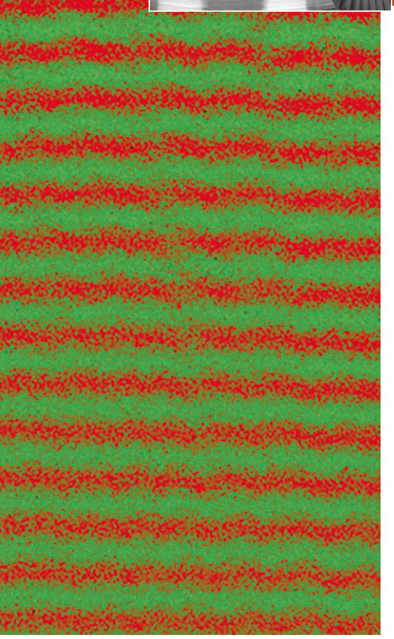

Commercialization of bulk nanostructured metals and alloys

Terry C. Lowe,* Ruslan Z. Valiev, Xiaochun Li, and Benjamin R. Ewing

Almost 30 years of research elucidating the mechanisms and reproducibility of nanostructuring has enabled the progressive emergence of reliable methods to manufacture bulk nanostructured metallic materials with superior properties. This article reviews examples of the use of nanostructured metals in engineered products that are currently commercially available, or will soon become available for specific biomedical, aerospace, electronics, and energy industry applications. The examples illustrate how the making and marketing of nanostructured materials follow similar development stages as other new advanced materials, but with additional challenges at each stage. Challenges include the difficulties of scaleup, intricacies of nanoscale characterization, the lack of consensus standards for product quality, competition with long-established conventional materials, regulatory hurdles associated with nanoscale technology, and consumer/user education on the virtues and limitations of nanostructuring. Finally, we discuss how the experiences to date with nanostructuring by various methods have established precedents that can guide manufacturing process development for advanced nanostructured metal and alloy applications.

Introduction

Nanoscale features in metals and alloys have existed throughout history, even in antiquity. However, the deliberate engineering of nanostructuring has resulted in superior properties and the knowledge to systematically control the processes that create nanostructures, especially in the past 10 years. Recent reviews[1,2] and articles published in this issue of the *MRS Bulletin* demonstrate examples of achieving high mechanical, physical, and chemical properties due to nanostructuring of metallic materials. At the same time, the commercial availability of nanostructured metals and alloys also depends on establishing suitable technologies and methods for manufacturing. We examine aspects of manufacturing to make nanostructured metals commercially available, focusing on issues and challenges that are unique to producing fine structured materials. Several examples are presented first to illustrate how nanostructured products are entering commercial markets.

Examples of commercialized nanostructured metals and alloys

Medical devices

Presently, metals and alloys are found in more than 70% of implanted medical devices.[3] Metallic materials provide excellent mechanical properties, plus adequate biocompatibility and durability. However, all of these characteristics of metals for medical devices are desired to be improved to enhance biofunctionality.

Included in the approaches to improve biofunctionality are the development of new alloy compositions, new manufacturing processes, and new surface modifications.[3,4] During the past two decades, nanostructuring metals by severe plastic deformation (SPD) has emerged as a new and auspicious approach to improve biofunctionality and mechanical properties.[1,2,5,6]

SPD processing techniques cause the formation of nanostructures both within bulk metals and alloys and on their surfaces. Thus, nanostructuring allows bulk alloys to subsequently be shaped or machined and also leave surfaces that enhance osteoblast adhesion.[7] Researchers have extensively explored the interaction of nanostructured alloys with physiological systems, which have been summarized in recent reviews.[2,8–10] The results of these investigations demonstrate benefits including increased cell adhesion, proliferation, and differentiation for a wide range of cell types. Most recently, Reiss et al. sequenced the RNA of bone-forming cells on nanostructured titanium to discover the mechanism by which the

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metal surface grain boundaries enhance osteoconduction.[10] The combination of enhanced biological properties with the superior mechanical properties of ultrafine grain alloys makes the pursuit of commercialization for trauma, orthopedic, and dental highly attractive.

This section describes several recent applications of such developments for medical practice.

Bone trauma fixation plate

Some of the earliest works on nanostructuring were applied to commercially pure titanium (CP Ti) because of its intrinsic biocompatibility with living tissues.[4] With the introduction of SPD processing to create nanostructured variants of CP Ti, it has become possible to develop medical implants with improved design due to the superior mechanical properties of nanostructured metals. For example, a mini-plate for bone fixation made of CP Ti, as specified by the ASTM F67 consensus standard, was used as the starting point for redesigning the product dimensions for making a nanostructured CP Ti version of the mini-plate. The cross-sectional properties of a new plate were computed using estimates of the fatigue endurance limit for coarse-grained and nanostructured Grade 4 Ti. **Table I** provides the estimation of the properties for the cross-sectional area of the nano-Ti plate.[11] The width and the diameter remained the same as in the standard item, whereas, due to the higher strength of the nanostructured CP Ti, the plate thickness was reduced from 0.9 mm to 0.7 mm. The plates were subjected to cyclic bending tests to measure their fatigue life. The standard plates sustained 17,000 ± 500 cycles while the redesigned plate made from nanostructured Ti survived a larger number of cycles to failure (105,000 ± 800). The plate made from nano-Grade 4 Ti possessed improved bending strength. This was one of the early examples that demonstrated the benefits of commercialization of such nano-Ti implants.

Dental implants

Nanostructured Grade 4 Ti offers multiple advantages for dental implants. Recently, nanoimplant dental implants have been produced from nanostructured Grade 4 Ti rods processed by the ECAP-Conform, a form of SPD processing.[12] The rods were machined to diameters of 2.0 mm, 2.4 mm, and 3.5 mm with the intraosseous part length of 8, 10, 12, and 14 mm by the company "Timplant" s.r.o., in the Czech Republic (http://www.timplant.cz/en/). An example of a 2.0-mm-diameter implant is shown in the x-ray radiographs in **Figure 1**. The panoramic x-ray image Figure 1a shows the placement of 2.0 mm nanoimplants in the narrow spaces between the left and right maxillary cuspids and central incisors. Figure 1b shows a magnified view of the small margins of bone tissue between the implant for the left lateral incisor and the adjacent teeth. Such a placement would not be feasible for larger diameter implants made from conventional titanium. Statistical analysis of records over two years from five surgeon dentists from state-owned and private dental surgeries in the Czech Republic has shown very good clinical performance of 2.4 mm nanoimplants.[13] Most dental implants have diameters of 2.9 mm or greater to sustain the high loads associated with chewing or clinching of the jawbone during stressful sleep. Because of the added strength of nanostructured Ti Grade 4, the small-diameter implants readily sustain these loads.

Incorporating nanoparticles in metals/alloys

Incorporating nanoparticles in metals and alloys breaks the barriers in traditional metallurgy and achieves significant performance enhancement that cannot be achieved by conventional methods.[14] One company, MetaLi, founded in 2016, started production in 2019, but already its nanotechnology-enhanced metal products are used by more than 100 companies/institutes in more than 20 countries worldwide (www.metaliusa.com). The practical applications of the nanotechnology-enabled solidification processing are growing rapidly and are expected to create widespread societal impacts, tremendous economic benefits, and significant energy and carbon savings.

One illuminating example of this technology is how it enables the welding of high-strength aluminum alloy AA7075, which has been considered difficult to join by fusion welding since its invention in the 1940s due to its hot crack susceptibility. Li et al. showed that AA7075 could be safely arc welded without hot cracks for the first time.[15] Nanostructured 7075 aluminum weld wire is shown in **Figure 2**. Joints welded with an AA7075 filler wire containing TiC nanoparticles exhibited fine globular grains and a modified secondary phase, which together helped eliminate the hot crack susceptibility. Moreover, the addition of TiC nanoparticles enhanced the tensile

Table I. Comparison of plate sizes for coarse grain and nano-CP titanium.

Plate	Fatigue Endurance Limit (MPa)	Geometrical Sizes of Mini-plate				Axial Resistance Moment (W)
		Length, L, mm	Area in the Central Part, A, mm^2	Thickness, h, mm	Width, b (mm)	$W = \frac{bh^2}{6}$ mm^3
Coarse grain Ti plate	340	46.4	2.7	0.9	3	0.40
Nano-Ti mini-plate	620	46.4	2.1	0.7	3	0.25

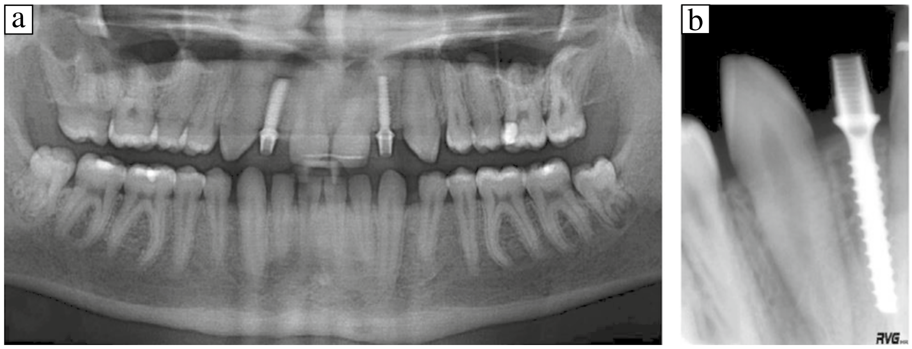

Figure 1. Nanoimplant with 2.0-mm diameter (www.timplant.cz/en/) from nanostructured Grade 4 Ti in a panoramic x-ray radiograph after surgery (a) and the control radiograph obtained after incorporation of the implant (b).

Figure 2. Nanostructured aluminum alloy 7075 weld rod/wire.

strengths in both the as-welded and post-weld heat-treated conditions.

Another example of enhancing bulk properties regard the tradeoff between mechanical properties and electrical conductivity in Cu with dispersed WC nanoparticles.[16] Nanoparticles can induce significant strengthening effects while having a less adverse impact on electrical/thermal conductivity than solute atoms. Cu–WC nanocomposites exhibit enhanced hardness/strength, Young's modulus, and thermal stability while maintaining high electrical/thermal conductivity. For example, such high level of properties of more than 600 MPa in tensile strength and 85% IACS in electrical conductivity were demonstrated in Yao, et al.[16] Cu–WC nanocomposites can potentially be employed for various applications that require high strength and high conductivity such as lead frames, electric motors, high-speed railway contact wires, heat exchangers, and electric resistance welding electrodes.

Introducing nanoparticles into metals and alloys during solidification is particularly useful for instilling continuous nucleation and controlling grain growth during conventional casting. A thermally activated nanoparticle dispersion mechanism in molten metals has been discovered, laying the foundation for the successful fabrication of metals containing dispersed nanoparticles by casting.[17] Hereby, bulk nanostructured metals via slow cooling have been developed, paving the way for the economical mass production of bulk samples with complex geometries. This discovery has great potential to significantly advance the commercial application of nanometals in aerospace, automotive, biomedical, electronics, and defense applications. However, for each prospective application, additional research is needed to customize the solidification processes.

Nanoparticle-enhanced alloy processing has the potential to impact the highest production volume family of alloys: steels.[18] Low-carbon advanced nanostructured steels can serve various structural engineering applications, from bridges, automobiles to stress-critical applications such as reactor pressure vessels in nuclear power stations. Since steels are among the lowest cost of all alloys, the economics of adding particles is critically important. Nevertheless, there are three major types of nanostructured steels that have been successfully commercialized.[19]

The first type is nanocrystalline bainitic steel. It solely relies on the solid-state phase transformation, which is accomplished by isothermal heat treatment of 15 days at 200°C, and therefore is unlimited by the size of the steel due to the lack of hot or cold plastic deformation. The second type is the nanostructured advanced high-strength steel (AHSS) produced by the Nanosteel Company. The nanostructured AHSS is produced by a combination of heat treatment and cold plastic deformation to achieve the refinement of grains and secondary phase. However, due to the necessity of cold plastic deformation, the nanostructured AHSS can only be produced in steel sheets. Nanosteel was founded in 2002 as a spinout of Idaho National Laboratory, operated by the US Department of Energy. The third type of nanostructured steel to be commercialized is oxide dispersion-strengthened (ODS) steel. The ODS steels are produced in powder form through ball milling steel powders with nano-sized oxide powders such as Al_2O_3 and Y_2O_3. Due to the limitation of the powder metallurgy process, the ODS steels have low production volume and high cost, and therefore are limited to small-scale applications. They are best known for their use in nuclear applications.

Challenges for nanostructured metals and alloys commercialization

The commercialization of nanostructured and most conventional advanced metals and alloys shares similarities and differences.

Scaling from long bar to coiled medical grade alloys for high-rate production

Nanostructured alloy production, which has already been established for long bars can be expanded to make coil for some medical devices and other demanding applications. Customers that presently purchase alloys in the form of coil expect the same product form for the economical delivery of nanostructured metals. As highlighted by Davis et al., a continuous-equal channel angular pressing (C-ECAP) process was shown to provide nanostructured alloys in coiled form.[20] Manufacturers with extensive experience in wire and small diameter bar production can readily augment existing conventional coil production to include nanostructured alloys that will provide higher performance for current and new customers. Coiling will be applied to high-strength variants of nanostructured grades of CP Ti, which have been produced with strength levels exceeding 1320 MPa and the fatigue endurance limit above 900 MPa.[21,22] These levels of mechanical properties for ultrafine grain commercially pure titanium outperform most titanium alloys.

Other variants of ECAP SPD processing have been implemented for titanium. For example, the CONFORM SPD process combines a single pass through a Conform SPD machine followed by rotary swaging.[23] This combination of processes is suitable for producing wires of diameter up to 1 mm with enhanced mechanical and fatigue properties.[22,24]

The superior properties of nanostructured alloys, mainly for medical applications, offset the higher prices of such materials. Other potential price-insensitive applications include luxury jewelry, where SPD-processed materials offer higher strength, hardness, and better wear resistance.

Scaling up to large volume production: Example of electrical conductors

Recent publications show examples of companies interested in enhanced metallic conductors to provide the means for transmission of electrical signals and energy in engineered systems from electronics to high-voltage power transmission cables. The energy efficiency in these applications is limited by the electrical conductivity of the conductor alloys. Commercial metals such as copper and aluminum possess the highest intrinsic conductivity, 59.6 MS/m and 37.7 MS/s, respectively. However, these levels of conductivity are rarely achieved in engineered products because of the need to add alloying elements or other treatments to enhance mechanical strength that, however, decrease electron mobility.

Large manufacturers have been evaluating nanostructuring by variations of equal channel angular pressing (ECAP) for copper[25–27] and aluminum[25,27–29] to enhance conductivity, strength, and other properties. For example, 5% IACS increases in electrical conductivity have been reported for ECAP of AA6xxx alloys.[8,29] At the same time, the strength is also increased. The achievement of both higher electrical conductivity and strength is attractive for applications such as overhead power transmission cables or replacing copper wiring in automobiles with aluminum.

A key to commercializing conductor alloys for applications demanding high-volume production is to demonstrate continuous production methods.[30] Most recently, coil-to-coil C-ECAP has been demonstrated for low-melting temperature alloys, including aluminum and magnesium.[20] Coiling has been applied to produce AA6101 and AA6201 conductor alloys up to 12 mm in diameter. High-speed operation of C-ECAP has been piloted and these results have been used to estimate annual production volumes within a large-scale manufacturing environment, including consideration of alloy yield efficiency, tooling changes, coil handling, and machine maintenance. For C-ECAP of 9.5 mm diameter rod at 0.6 m/s with 96% production yield efficiency, a single C-ECAP system can produce 49.7 km of coiled aluminum conductor alloy per day, corresponding to 9.5 metric tons/day. Thus, for automotive conductor applications, a single C-ECAP system can produce an estimated 13,700 km/day of 130-μm diameter, 19-strand conductors. For high voltage overhead power lines, a single C-ECAP machine can produce 105 km/day of aluminum conductor steel-reinforced (ACSR) sparrow cable (six aluminum strands, each 2.67 mm in diameter) or 12.5 km/day of Osprey cable (18 aluminum strands, each 4.447 mm in diameter). Thus, high-volume nanostructuring is feasible but integration of the nanostructuring process with other steps in manufacturing is necessary to realize the true economies of scale. This integration has not yet been demonstrated for aluminum conductors.

The substitution of nanostructured aluminum conductors for copper in automobiles is another compelling example because of the 1.65-fold weight savings per delivered amp, the 1.5-fold lower cost per amp, and the enhanced fuel economy associated with the weight reduction. Furthermore, reducing the amount of copper wiring in automobiles increases the extent to which automobile steel can be recycled without contamination from copper. Companies in Europe, North America, and Asia are exploring the advantages of nanostructured conductor alloys. However, the adoption of nanoscale materials is impacted by the economic necessity for high-volume production to achieve economies of scale. While foundational technologies such as coiling have been demonstrated, the integration of continuous casting of aluminum with subsequent nanostructuring at the rates that conventional alloys are produced has yet to be demonstrated. Such demonstrations generally develop over many years of refinement of manufacturing practices.

Long-term stability

The fine-scale structures in nanostructured metals that impart superior properties also can create issues for long-term stability. Thermodynamic driving forces to reduce free energy in all material systems favor the gradual elimination of the same microstructural features that underpin the superior properties of nanostructured metals. For example, processes to reduce grain-boundary area, decrease dislocation densities, and eliminate nonequilibrium structures can result in issues with long-term stability. The introduction of nanoparticles, as described in the previous example, illustrates an approach to ensure long-term stability. However, uncertainty at the outset about long-term performance can be an impediment to the commercialization of nanostructured metals in some applications. Only after many years of documented performance can nanostructured metals be considered for some applications, such as long-term medical implants. Thus, the adoption of nanostructured metals will be deliberately delayed for applications in which long-term stability must be known in advance.

Nanoscale characterization

Ironically, advances in characterization techniques such as atomic force microscopy, three-dimensional atom probe tomography, or electron backscatter diffraction by transmission kikuchi diffraction are partly responsible for enabling the development of nanostructured metals and alloys. Such techniques have enabled the careful characterization of nanostructures and the development of the processes by which they can be formed. For commercialization, the use of such techniques is problematic and uneconomical. For example, manufacturers of metal products invariably need to inspect the metals and alloys they receive. This can be problematic when inspection of nanostructured metals requires advanced characterization techniques. For example, a basic inspection of standard material parameters such as grain size is typically performed via optical microscopy. Yet, the conventional methods of inspecting incoming alloys using optical microscopy are ineffective for nanostructured metals since the grains can be smaller than can be resolved with white light. Furthermore, the equipments needed to characterize nanostructured metals are expensive and are rarely available in manufacturing environments. Also, personnel with the level of education (graduate level) needed to operate and interpret data from advanced characterization tools such as electron microscopes are often unavailable in manufacturing environments. Consequently, it can be difficult to adopt nanostructured metals in commercial applications without investing in high-end characterization equipment and hiring experienced researchers with graduate degrees. The personnel and equipment needed to conduct the elaborate techniques needed to document microstructures upon receipt of nanostructured metals are beyond the reach of many industries.

Even mechanical testing of nanostructured metals and alloys can be problematic. High-strength materials require special considerations to be tested mechanically. Notch sensitivity is particularly problematic and requires special attention to sample mounting and alignment during tensile testing. Standard knurled-surface wedge grips for mounting tensile samples can induce failure of nanostructured metals within the grips.

Frequently, hardness testers are used in industrial environments to obtain a rough approximation of strength. Hardness tests measurements, such as Rockwell or Brinell hardness testing, are commonly correlated with ultimate tensile strength for many conventional alloys. However, hardness and tensile strength do not correlate as consistently for nanostructured alloys. Thus, this established, quick method to approximate tensile strength is not as useful for nanostructured metals.

Consensus standards

In many industries, products and manufacturing processes must satisfy consensus standards such as ISO, DIN, ASTM, ANSI, and others. Yet, for some nanostructured alloys, standards do not yet exist. Consequently, products for highly regulated industries such as aerospace and medical devices are slow to adopt nanostructured metals, unless they are adequately covered by existing standards. While standard developing organizations worldwide have formed nanotechnology-related committees to review and approve standards for nanoscale engineered materials and products, their emergence lags the industries that are ready to adopt the benefits of nanostructured metals and alloys.

Competition between conventional and nanostructured metals

The most significant barrier to any new innovative product is the presence of the existing, and often long-established, conventional competing product. The improvements in metals and alloys are typically incremental, for example, 5% to 10% improvements over an existing product. But for nanostructured metals and alloys, the improvements in properties can be 30% to 200% beyond existing products. Uncertainty, and even disbelief, about how such improvements are even possible, creates a barrier to their adoption. Familiarity with the status quo often dominates the choice of materials. Adopting intricate designs or elegant mechanical engineering is easier to perceive and understand, such that design innovations are often favored over material innovations such as nanostructuring.

Regulatory impacts

The use of advanced materials in some applications is subject, not just to meet consensus standards, but also industry-specific regulations. In transportation industries (automotive, aerospace, rail, marine), government-specified regulations for safety, environmental protection, and energy efficiency must be met. For instance, most nations have highway traffic safety regulations. New nanostructured materials used in automobiles must be proven to meet these regulations. Such testing tends to delay the introduction of nanostructured alloys. However, some regulations, for example, the Corporate Average Fuel Economy (CAFE) standards in the United States, create a motivation for rapid adoption of nanostructured metals. For example, a nanostructured alloy with 50% greater strength than its conventional alloy counterpart will permit the use of less metal for the same function, thus decreasing weight and increasing fuel efficiency. Similar regulations encourage the automobile industry to reduce the weight of vehicles in the European Union (EU). Regulation (EC) 443/2009 targets fleet-wide average emissions of 95 g CO_2/km, which corresponds to fuel consumption of 4.1 l/100 km. Since 2019, a penalty of €95 has been applied per each g/km of exceedance for each registered vehicle, providing a substantial financial incentive for adopting higher-performance materials such as nanostructured alloys.

For medical product applications, any substitution of a new material requires extensive validation to prove that the substitution results in improved performance to achieve regulatory approval. Thus, new nanostructured alloys to conventional alloys undergo comparable scrutiny. However, nanostructured alloys may have an advantage in that alloys with previously approved chemical composition can be nanostructured to enhance performance, but without the additional risks posed by introducing new chemical compositions. This simplifies the regulatory approval processes. Similarly, simple single-phase alloys such as Ti-15 wt.% Zr can be nanostructured to achieve high strength, whereas other advanced alloys depend upon more complex alloy formulations, which because of their complexity are less attractive for medical applications.

Consumer education and limitations

Prospective adopters of nanostructured metals value their enhanced properties, but sometimes fail to understand the limitations of nanostructuring. While nanoparticles can enhance weldability of some alloys, as previously described, nanostructures in metals, without nanoparticle reinforcements, cannot be joined by fusion welding. Welding generally eliminates the nanoscale structures that have been imparted by SPD or other methods. Thus, products that are shaped into their final form only by machining, or other low-temperature processes, are best suited for nanostructured metals.

Another area in which greater knowledge by consumers and manufacturers is needed regards the cost to purchase and use nanostructured metals and alloys. Because additional steps are required to impart fine-scale structures in metals, it is appropriate to expect increased costs to manufacture nanostructured materials. However, other factors may offset higher production costs. For example, studies have shown that nanostructured metals have greater machinability and formability than their conventional counterparts.[31,32] Thus, the total cost to manufacture products for which extensive machining is required may be decreased by using nanostructured alloys. Similarly, the formability of nanostructured alloys has been shown to be superior to conventional alloys, thereby reducing the cost of production and also improving resistance to in-process failure.[33,34] There is a need for educating consumers on these tradeoffs, especially since the advantages and limitations of nanostructured metals differ between alloy families. Generalized claims of benefits of nanostructuring have the potential to mislead prospective adopters of nanostructured metals. Specific, easily understood evidence of the advantages and disadvantages needs to be available to inform nonexperts about nanostructured metals and alloys.

Summary

The challenges of introducing nanostructured metals are being addressed by universities, institutes, and companies worldwide. Examples of nanostructured metal products show commercial viability, at least for small-volume production. Greater economies of scale are needed for high-volume production. Early examples highlighted here include two nanostructured titanium medical implants and two metal matrix nanocomposites, AA7075 aluminum–titanium carbide nanocomposite welding filler wire and copper–tungsten carbide nanocomposite electrical conductors. Commercialization of nanostructuring was also highlighted for bainitic steels, advanced high-strength steels, and oxide dispersion-strengthened steels.

Specific challenges to commercialization need to be addressed, including providing better consumer education, navigating regulatory impacts and barriers, developing suitable low-cost characterization techniques, assuring long-term stability, and developing more effective methods for processing nanostructured metals. The examples discussed point to various stages of market impact. So far, nanostructured metals have not yet displaced conventional alternatives. However, as commercial product offerings grow, it is reasonable to expect accelerating incorporation of nanostructured metals and alloys into engineered products.

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Conflict of interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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