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Magnesium Technology

Steven Barela
Aeriel Leonard
Petra Maier
Neale R. Neelameggham
Victoria M. Miller







Steven Barela • Aeriel Leonard • Petra Maier • Neale R. Neelameggham • Victoria M. Miller Editors

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TMS



Editors
Steven Barela
Terves Inc./Magnesium-USA
Euclid, OH, USA

Petra Maier University of Applied Sciences Stralsund Stralsund, Germany

Victoria M. Miller University of Florida Gainesville, FL, USA Aeriel Leonard The Ohio State University Columbus, OH, USA

Neale R. Neelameggham IND LLC South Jordan, UT, USA

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Preface

Magnesium (Mg) and its alloys continue to be extensively investigated for applications in the automotive and aerospace industries as well as biomedical. Although the largest use of Mg is desulfurization of steel, followed by alloying of aluminum, its primary attributes as the lightest structural metal include high strength-to-weight ratio, good vibration damping, electromagnetic and RF shielding, low toxicity, and controllable corrosion rates for dissolvable applications. It is because of these attributes that Mg will play a crucial role in reducing the carbon footprint and forwarding the development of sustainable technology, specifically in the replacement of conventional materials such as steels and aluminum alloys. Compared to carbon fiber composites, Mg is more cost effective and can be readily recycled and offers metallic ductile behavior.

Collaborative research and development by researchers, scientists, engineers, industry, government agencies/laboratories, and academic institutions are actively working to develop roadmaps for next-generation products and address these challenges through innovative alloy designs and methods. The TMS Magnesium Committee has been actively involved in providing a platform for these institutions to disseminate the latest information, developments, and cutting-edge research and development, and to present the latest research and development trends related to magnesium and its alloys through the Magnesium Technology Symposium held each year at the TMS Annual Meeting & Exhibition.

The twenty-fourth volume in the series, *Magnesium Technology 2023*, is the proceedings of the Magnesium Technology Symposium held during the 152nd TMS Annual Meeting & Exhibition in San Diego, California, March 19–23, 2022. The volume captures papers and extended abstracts from 12 different countries. The papers have been categorized based on topics pertaining to alloy design, fundamentals of plastic deformation, primary production, recycling and ecological issues, characterization, joining, machining, forming, degradation and biomedical applications, corrosion and surface protection, and computational materials engineering.

The symposium began with keynote sessions that featured several distinguished invited speakers from industry, government organizations, and academia, who provided their perspectives on the state of the art, goals, and opportunities in magnesium alloy research and development. Andrew Sherman from Terves Inc./Magnesium-USA spoke about defending patents/intellectual property. Aaron Palumbo of Big Blue Technologies addressed reductant consideration in thermal pathways to primary magnesium metal production. Carolyn Woldring, co-founder of Magsorbeo Biomedical, discussed engineered bioabsorption for medical implant applications. Mr. Michael Ren, Sunlightmetal Consulting Inc., spoke about primary Mg production, specifically development of compound-vertical-retort technology for magnesium production and its application. Norbert Hort with Helmholtz-Zentrum Hereon, Geesthacht, Germany spoke about the concept of metastable vs. stable. Mohammadreza Yaghoobi with the University of Michigan covered recent advances in PRISMS-Plasticity software for simulation of deformation in Mg alloys. Mariyappan Arul Kumar from Los Alamos National Laboratory gave a presentation about the combined effect of alloying and grain size on the deformation behavior of magnesium alloys.

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In conclusion, the 2022–2023 Magnesium Committee would like to thank and express its deep appreciation to all authors who contributed to the success of the symposium; our panel of distinguished keynote speakers for sharing the newest developments and valuable thoughts on the future of magnesium technology; all the reviewers for their best efforts in reviewing the manuscripts; and the session chairs, judges, TMS staff members, and other volunteers for their excellent support, which allowed us to develop a successful, high-quality symposium and proceedings volume.

Steven Barela Aeriel Leonard Petra Maier Neale R. Neelameggham Victoria M. Miller

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About the Editors



Steven Barela is from Pueblo, Colorado, home to the Colorado Fuel & Iron Steel Mill (now Evraz). Driven by the need to resolve welding problems encountered when fabricating race cars, he attended the University of S. Colorado (now CSU-Pueblo) earning an A.A.S. in Metallurgical Engineering Technology. This led to a position at Rocky Flats/DOE nuclear assembly facility in the Non-Nuclear Joining R&D group as an Intern Engineer while simultaneously earning a B.S. in Metallurgical & Materials Engineering at the Colorado School of Mines (CSM) where he specialized in joining and was involved in the CSM Joining Research Center. Mr. Barela then went on to work at Martin Marietta Astronautics Group in the Advanced Manufacturing Technology Group which oversaw procedures and the production of the Titan family of launch vehicles, specifically all joining operations (braze, TIG, MIG, Variable Polarity Plasma) on aluminum, stainless steel, titanium, and metal matrix composites materials. He also participated in the development of Weldalite, a program to produce the Al-Li external tank for the NASA space shuttle program. In the late 1990s, Mr. Barela transitioned his career from welding metallurgy technology to marketing, product development, and sales of welding, forging, and fabricated products as a Technical Sales Engineer at Timminco Extruded Magnesium Products. He then worked for 10 years for Solikamsk Magnesium Works (Russia) by running the U.S. subsidiary Magnesium.com, Inc. During his tenure, Mr. Barela championed forged Magnesium (Mg) wheel projects, generating, and overseeing sales of various Mg products worldwide. Currently, Mr. Barela is with Terves Inc./Magnesium-USA overseeing marketing, product development, and technical sales for extruded and forged products. Over the 20+ years as a member of the TMS Magnesium Committee, Mr. Barela has brought practical industrial end-use knowledge, experience, and insight to the proceedings.

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Aeriel Leonard is an Assistant Professor in the Materials Science and Engineering Department at The Ohio State University. She was awarded the Department of Energy Early Career Award in 2022 and the Office of Naval Research Young Investigator Award in 2021. She earned her B.A. in Metallurgical and Materials Engineering from the University of Alabama in 2012. In 2013, she began her Ph.D. journey at the University of Michigan in Materials Science and Engineering where she earned her Ph.D. in 2018. Dr. Leonard's Ph.D. work investigated real-time microstructural and deformation evolution in magnesium alloys using advanced characterization techniques such as high energy diffraction microscopy and electron back scatter diffraction. During her time at the University of Michigan, she led and worked on many teams aimed at increasing the number of underrepresented minorities in engineering including developing and implementing a leadership camp for female engineering students in Monrovia, Liberia. Dr. Leonard was awarded an NRC Postdoctoral Fellowship at the US Naval Research Laboratory in Washington, DC where she worked for two years. During this time, she used advanced characterization techniques such as x-ray computed tomography and high energy diffraction microscopy to understand damage and texture evolution during in-situ loading in additive manufactured materials. She also runs a lifestyle blog titled Aeriel-Views aimed at young graduate and professional students.



Petra Maier received her doctoral degree from Loughborough University, United Kingdom, in 2002 in Materials Science, in the field of grain boundary segregation in steel. After completing her Ph.D., she worked at the University of Applied Sciences Wildau, Germany, as a postdoctoral fellow under the supervision of Prof. Asta Richter with a focus on mechanical properties by nanoindentation. From 2004 to 2006 she worked as a research associate in the MagIC under the supervision of Dr. Norbert Hort at the Helmholtz-Zentrum Geesthacht, Germany, being a part of the Institute of Materials Research lead by Prof. Karl Ulrich Kainer. Her focus was on magnesium recycling and high-temperature alloy development.

From 2006 to 2008, Dr. Maier was a research associate at the Technical University Berlin, Germany, in the Institute of Material Sciences and Technologies, Department of Materials Engineering of Prof. Claudia Fleck. There, her research specialties included corrosion fatigue on magnesium.

Since 2008, Dr. Maier is a professor of Materials and Production Engineering in the School of Mechanical Engineering at the University of Applied Sciences Stralsund in Germany. Since October 2022, she is a Lise Meitner professor at Lund University in the Department of Mechanical Engineering Sciences. She enjoys working in the field of Mg-based biodegradable implants. Her research is focused on corrosion under stress, the influence of the corrosion morphology on

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mechanical properties and how crack initiation and propagation is influenced by the microstructure. She is currently a past-chair of the TMS Magnesium Committee.

Neale R. Neelameggham IND LLC, is involved in international technology and management licensing for metals and chemicals, thiometallurgy, energy technologies, Agricoal, lithium-ion battery, energy efficient low cost OrangeH2, Netzero sooner with Maroon gas and Pink hydrogen, rare earth oxides, etc. He has more than 38 years of expertise in magnesium production and was involved in the process development of its startup company NL Magnesium to the present US Magnesium LLC, UT until 2011, during which he was instrumental in process development from the solar ponds to magnesium metal foundry. His expertise includes competitive magnesium processes worldwide and related trade cases. In 2016, Dr. Neelameggham and Brian Davis authored the ICE-JNME award-winning paper "Twenty-First Century Global Anthropogenic Warming Convective Model." He is working on Agricoal® to greening arid soils, and at present energy efficient Orange hydrogen, and methane abatement. He authored the ebook The Return of Manmade CO₂ to Earth: Ecochemistry. Dr. Neelameggham holds 16 patents and applications and has published several technical papers. He has served in the Magnesium Committee of the TMS Light Metals Division (LMD) since its inception in 2000, chaired in 2005, and since 2007 has been a permanent advisor for the Magnesium Technology Symposium. He has been a member of the Reactive Metals Committee, Recycling Committee, Titanium Committee, and Program Committee for LMD and LMD council. Dr. Neelameggham was the Inaugural Chair, when in 2008, LMD and the TMS Extraction and Processing Division (EPD) created the Energy Committee and has been a Co-Editor of the Energy Technology Symposium through the present. He received the LMD Distinguished Service Award in 2010. As Chair of the Hydrometallurgy and Electrometallurgy Committee, he initiated the Rare Metal Technology Symposium in 2014 and has been a co-organizer to the present. He organized the 2018 TMS Symposium on Stored Renewable Energy in Coal.

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Victoria M. Miller is an Assistant Professor in the Department of Materials Science and Engineering at the University of Florida, a position she started in September 2019. She was previously an assistant professor at North Carolina State University from 2017 to 2019. Originally from Michigan, she received her B.S.E. in Materials Science and Engineering from the University of Michigan in 2011 and completed her Ph.D. in Materials at the University of California Santa Barbara in 2016.

After graduate school, she worked for a year at UES, Inc. onsite in the Materials and Manufacturing Directorate of the Air Force Research Laboratory in Dayton, Ohio. She also previously worked at Ford Motor Company, Toyota Engineering and Manufacturing, and Lockheed Martin Aeronautics.

Her primary research interest is microstructural evolution during thermomechanical processing of metals and alloys, particularly for those with low symmetry crystal structures. She has been researching Mg alloys since the age of 16.

Professionally, Dr. Miller has served on many committees within TMS, is Associate Editor for *JOM*, and is a Key Reader for *Metallurgical and Materials Transactions A*. She was a recipient of the 2017 TMS Young Leaders Professional Development Award, the 2020 ASM Bronze Medal Award, and the 2022 TMS-JIMM Young Leaders International Scholar Award.

Session Chairs

Magnesium Technology

Keynote Session

Steven Barela, Terves Inc./Magnesium-USA Petra Maier, University of Applied Sciences Stralsund Aeriel Leonard, The Ohio State University

Microstructure Evolution

Benjamin Anthony, University of Florida Kiran Solanki, Arizona State University

Corrosion and Coatings

Petra Maier, University of Applied Sciences Stralsund Joshua Caris, Terves Inc.

Deformation and Advanced Processing

Domonkos Tolnai, Helmholtz-Zentrum Hereon Vineet Joshi, Pacific Northwest National Laboratory

Primary Production and Recycling/Alloy Development

Aaron Palumbo, Big Blue Technologies

Reviewer Pool

Benjamin Anthony, University of Florida, USA Christopher D. Barrett, Mississippi State University, USA Benjamin Begley, University of Florida, USA Tracy Berman, University of Michigan, USA Jan Bohlen, Magnesium Innovation Centre, Germany Joshua Caris, Terves Inc., USA Hajo Dieringa, Helmholtz-Zentrum Hereon, Germany Jarek Drehlich, Michigan Technological University, USA Adam Griebel, Fort Wayne Metals, USA Daniel Höche, TU Darmstadt, Germany Norbert Hort, Helmholtz-Zentrum Hereon, Germany Steven Johnson, Central Connecticut State University, USA Brian Jordon, Baylor University, USA Vineet V. Joshi, Pacific Northwest National Laboratory, USA Alan Luo, The Ohio State University, USA Petra Maier, University of Applied Sciences Stralsund, Germany Michele Manuel, University of Florida, USA Suveen Mathaudhu, Colorado School of Mines, USA Victoria Miller, University of Florida, USA Neale Neelameggham, IND LLC, USA Aaron Palumbo, Big Blue Technologies, USA Regine Willumeit-Römer, Helmholtz Center Hereon, Germany

Part I Keynote Session



Sustainable Domestic Manufacturing and Protecting IP in a Post AIA World

Andrew Sherman

Abstract

Ten years ago, the American Invents Act was passed by Congress. Despite lofty intentions, the AIA significantly degrades the value of IP and patents. Terves has recently gone through an enforcement action against a large importer of foreign magnesium products. The current system is rigged against small company inventors, with non-technical administrative judges invalidating 84% of patents in favor of infringers, versus <50% by multiple skilled patent examiners during reexaminations. Enforceable IP is essential for American competitiveness to complete with subsidized, unregulated, and lower cost offshore locations. Terves is a member of the US Inventor, representing 60,000 inventors focused on restoring individual patent rights post AIA. Terves' experience enforcing IP rights in today's climate as well as US inventors pending bill to restore patent rights will be discussed along with potential strategies and actions that inventors can take to mitigate AIA limitations.

Keywords

Magnesium • Intellectual property • US inventors

Background

Middle market materials manufacturers face stiff competition for overseas competitors that have unfair advantages in labor, regulatory, energy, and capital costs as a result of operating out of non-free market economies. In order to recreate a domestic manufacturing industry for critical materials, including magnesium, public policy and investment is required to offset these substantial disadvantages.

A. Sherman (\omega) Terves LLC, Euclid, OH, USA

e-mail: asherman@tervesinc.com

Recent developments include investment and production tax credits, proposed rebates/credits for domestic sourced materials, enhanced buy-American and domestic content requirements, and public investment (loans, grants) to reduce and spread capital risk. Intellectual property, including patents, trade secrets, and trademarks (branding) can also establish a barrier to offshoring. However, the American Invents Act (AIA) has changed the patent landscape, making enforcement more difficult and expensive.

Introduction to PMT Group and Terves LLC

PMT group is a group of critical materials manufacturers, including Terves LLC, which is North America's only vertically integrated producer of wrought magnesium. Parent Company Powdermet Inc. was formed in 1996, when it acquired the powder metallurgy assets of refractory materials producer Ultramet Inc. Powdermet and its affiliates still provide powder metal and cermet feedstocks and toll powder production services for highly engineered materials. In 2013, Terves LLC was formed to commercialize "engineered response" materials which provide tailored responses to the environment, including changing dimensions (expanding), disintegrating, generating heat, releasing chemicals, or producing a signal. In 2016, to meet demand and reduce costs for domestically produced TervalloyTM dissolvable magnesium alloys, Terves built a permanent mold foundry, added a 4000 ton extrusion facility and CNC machine shop, representing a significant investment in critical materials and magnesium production. Today, Terves has established capacity for over 1000 tons/annum of critical materials (Mg) production, and has over 40 patents on numerous magnesium alloys and engineered response materials and their applications. In addition to extruded products and powder metal and cermet feedstocks, Terves and Powdermet also provide metal matrix composite materials, forging

stock, fully machined parts to customer specifications, and full assemblies built to OEM specifications.

As part of our efforts to diversify markets and products, in 2021, Terves commissioned Wagstaff to design and construct North America's only magnesium vertical direct chill (VDC) casting system to be built in the last 35 years. With a capacity of 3000-5000 tons/annum of magnesium billets, Terves can support additional markets in recreation/sporting goods, defense, aerospace, biomedical, energy storage, and transportation lightweighting markets. To support these markets, Terves has developed and licensed a number of new heat treatable Magnesium alloys, including new magnesium alloys specifically engineered for high plasticity (energy absorption), high strength, lower cost (lower alloy content and higher extrusion, forming, and rolling speeds), and with reduced or eliminated high value rare earth contents. When the new VDC comes on-line (spring, 2023), the current permanent mold foundry will be converted to the metallothermal production of rare earth metals, including Nd, Gd, and Dy. Parent company Powdermet Inc. also broke ground in August, 2022 on a new 30,000 sq ft facility to produce MnBi (licensed from the critical materials institute) rare earth free magnets, as well as NdFeB rare earth magnetic materials via strip casting using purified, recycled, or internally reduced rare earth metals.

In 2014, Terves introduced its patent pending TervalloyTM dissolvable magnesium alloys. This development transformed the oil and gas completions industry, reducing water use and emissions during completion operations by up to 92% by eliminating the need to drill out plugs while flushing the produced debris from the 3-5 mile well-string in order to reestablish communication with the geologic formation. Plugs made from Tervalloy simply dissolve/ disintegrate upon exposure to fluid, salinity, and temperature, returning the magnesium to the seawater from which it was originally extracted (Mg is 2% of seawater) in a controlled manner. By 2017, when our first patents issued, the majority of the market had been offshored to low cost Chinese suppliers who use the pigeon process (high CO₂) and low cost labor, utilities, and regulations to out-compete western manufacturers. Magnesium has been designated as a critical material for lightweighting (including its use in aluminum alloys, titanium production, and steel processing) primarily due to unfair Chinese trade practices (https://doi. org/10.3133/ofr20181021).

Patents are one strategy that act as a sword to attack infringers, including unfair foreign competitors. Terves has been working to enforce its patents, and after tracking a large importer of infringing magnesium alloys, we filed for patent enforcement/infringement in 2018. *Terves LLC v. Ecometal Inc.*, Case No. 1:19-cv-1611-DCN (N.D. Ohio 2019). After more than three years, Terves prevailed at trial, and after surviving two IPR's and an ex-party reexamination, the

patent validity, infringement, and lost profit damages were established in district court, and a permanent injunction was issued against the infringing parties. Terves continues to enforce its patents to sustain North American manufacturing and is increasing its investment and commitment to critical materials production in the USA with a planned \$27 M investment in expansion and additional production at our Euclid, Ohio facilities.

Patents, IP, and Enforcement Post AIA

The right to a patent was granted to authors and inventors in the US constitution. It was the first time in history that the common "man or woman" were allowed to own their inventions. Prior to the US constitution, grants were by the state, generally to landed and gentry class, and the common person had no right to ownership at the national level. To support sustainable domestic manufacture of critical materials, PMT group has over 100 patents issued or pending and has invested heavily in IP creation and enforcement as a core strategy to create barriers to (mainly) foreign competition and to try and prevent rapid commoditization of products to allow faster payback and acceptable returns on investment in the capital intensive and long product life cycle materials industry. After 26 years, (including several prior freedom to operate, patent infringement, and theft of trade secret litigations), the current enforcement experience has highlighted the strengths and weaknesses, and expenses, of IP strategy. In particular, the patent trial and appeals board (PTAB), created under the AIA, and set up by big tech, has overturned 85% of all patents brought before it, using administrative court judges with no requirement for any patent or technology expertise. The PTAB was designed with that in mind by big tech. In fact, Googles' main patent strategist, Michell Lee, was appointed head of the patent and trademark office by president Obama and instituted the rules and procedures designed to greatly weaken patent holders rights and remedies, and to shield large corporations from patent infringement damages. Ostensibly, the PTAB was supposed to be a faster and cheaper method of resolving patent validity as compared to district court proceedings. In reality, it was designed in favor of large corporations to allow them to utilize other peoples intellectual "property" compensation.

We are part of US Inventor, an organization of over 60,000 inventors working to restore patent rights that were largely stripped by the PTAB under the American Invention Act (AIA). According to the US Inventor website "The America Invents Act of 2011 (AIA) created an easier way to invalidate (revoke) an issued patent. The PTAB is an administrative court with no jury and much less due process than a real court. Rather than a lifetime-appointed judge, a

PTAB trial typically has three attorneys who are called Administrative Patent Judges (APJs). Since inception, 84% of the patents that go through a PTAB process get fully or partially invalidated (partially usually means the parts of the patent that matter).

When you attempt to stop a large corporation from infringing your patent, they will try to use the PTAB to invalidate your patent. If you win one PTAB attack, you can still be pulled into additional ones by the same or other infringers. According to the AIPLA (American Institute of Patent Law Association), a reasonable PTAB defense costs \$400,000 to \$800,000. Historically, the typical inventor would hire an attorney on a contingency basis to fight an infringer (where the inventor doesn't pay much up front and the attorney gets a percentage of the verdict award or settlement amount). Today, it is extremely rare for an attorney to take any PTAB case on contingency."

Furthermore, the AIA largely removed the ability to stop infringers, we believe against the constitution (Supreme Court ruled that patents are no longer a property right, to allow the PTAB to exist). Per US Inventor "The U.S. Supreme Court decided that it was in the "public interest" for a proven infringer to continue infringing because it could serve the market better than a startup (Ebay, 2006). As a result, even if you win your case, you will have to pass a "public interest" test before an injunction can be issued to stop the infringer. A startup vs an entrenched corporation will typically fail this test, so you can't stop the infringer. You end up with a court-ordered royalty that you cannot negotiate, and the infringer keeps your invention and the market. This is often an impossible barrier for what would have been, until recently, the next great American disruptive startup."

US Inventor is working to restore US Inventor rights, including the ability of inventor-operators to opt out of the IPR in favor of district court (IPRs are almost exclusively filed in response to a claim of infringement), and the restore the right no not be forced to license to a corporation that has been found to infringe.

Terves' experience in litigating was that it was long (over three years), mainly due to the ability of the infringer to delay and stall litigation and particularly discovery. We were unable to serve or get discovery in china, and have abandoned our Chinese patents as worthless due to their extremely poor legal system (no experts, no discovery, mainly works on behalf of government interests, not private party interests). Cost of litigating, including defending two IPR's and one reexamination, discovery, motions, and trial approached \$2M. We did receive a permanent injunction and lost profits, proving to the jury that no reasonable alternative to Tervalloy is available in the market, a major win.

One of Terves strategies to offset the power of the PTAB has been to obtain a large number of patents and claims, to

increase costs of litigation against our IP portfolio. Terves currently has more than five patents issued and over 350 claims issued on our dissolvable magnesium materials and their application. However, this comes at significant cost to both prosecute (obtain), defend (against claims of invalidity), and enforce (attack infringers). This is in addition to substantial investment in vertical integration, inventory, and productivity to reduce costs, speed delivery, and meet all demand for wrought magnesium in North America. We also invest and collaborate to develop new alloys, heat treatments, and production processes to enhance performance, customize products for individual clients, and meet customer demands in order to better compete with foreign producers and importers.

In addition to our district court enforcement actions, we have been researching ITC 337 exclusion processes. These have been used by some startups (Aspen Aerogels, for example) to block import of infringing product. The advantage of 337 proceedings is they cannot be stalled by legal maneuverings, and reach conclusion in 18 months or less under statutory requirements, leading to an exclusion order blocking imports of infringing products into the USA. Disadvantages are that the costs are high and cannot be recovered, nor can damages be recovered, and the exclusion order needs to be enforced by an overworked customs and border patrol. However, violators of the exclusion order are subject to punishing fines and penalties that can reach \$100,000/day, and shipments and product can be confiscated. Those that have successfully pursued 337 have indicated that they largely rely on the risk aversion of large public users to enforce and comply, in addition to working with customs and border patrol to identify and seize inbound infringing products. This was successful in the case of Aspen Aerogels, while it was unsuccessful in the case of crucible materials, who won a general exclusion order for NdFeB magnets but did not prevent offshoring (no magnets are currently produced in the USA, despite the fact that they were invented and at one time solely produced in the USA). Many of the ITC procedures and precedents were set in the crucible materials action, and ITC proceedings are increasingly being used to enforce and enhance patent rights for composition of matter and other utility patents.

Summary

In summary, patents, trademarks, and trade secrets are one set of tools to support domestic manufacturing. Innovation, particularly process improvements to reduce costs and labor content as well as creating new markets/demand, are more powerful in the short run, but patents provide for longer term sustainability, but only if they can be successfully enforced and defended. Novel composition of matter patents are the

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easiest to defend and enforce (as in the Tervalloy case, we enforced composition of matter claims). Branding, including trademarks, is an incredibly powerful and relatively easily enforced tool to create a preference and loyalty but is more difficult to achieve in the materials and manufacturing sector serving OEM's, as opposed to the consumer sector. Copywrites, including material performance specifications,

quality documents, and validated property datasets, can also create preferences and increase cost of competition in critical applications. Finally, government intervention and public policy efforts such as domestic content rules, tariffs, tax incentives, and targeted investments are required to offset the actions of foreign governments and non-free market economies to create fair trade, as opposed to free trade.