REPORT DOCUMENTATION PAGE				Form Approved OMB NO. 0704-0188			
The public reporting burder searching existing data sou regarding this burden estin Headquarters Services, Di Respondents should be awa of information if it does not of PLEASE DO NOT RETURN	n for this collection of in rces, gathering and mai mate or any other aspe rectorate for Information are that notwithstanding lisplay a currently valid C YOUR FORM TO THE	nformation is estimated to ntaining the data needed, ect of this collection of i n Operations and Report any other provision of law, MB control number. ABOVE ADDRESS.	o avera , and co nformat ts, 121 , no per	ge 1 hour per ompleting and tion, including 5 Jefferson D son shall be su	respon eviewir sugges avis Hig bject to	se, including the time for reviewing instructions, ing the collection of information. Send comments stions for reducing this burden, to Washington ghway, Suite 1204, Arlington VA, 22202-4302. any oenalty for failing to comply with a collection	
1. REPORT DATE (DD	-MM-YYYY)	2. REPORT TYPE			3.	DATES COVERED (From - To)	
04-03-2015		Final Report				5-Dec-2011 - 4-Dec-2014	
4. TITLE AND SUBTI	ГLE			5a. CO	5a. CONTRACT NUMBER		
Final Report: Resear	ch Area 10: Synth	esis and Processing	g of	W9111	W911NF-12-1-0012		
Materials: Enhancing Deformation Processing Capability of Structural HCP Metals				5b. GR.	5b. GRANT NUMBER		
					5c. PROGRAM ELEMENT NUMBER		
6 AUTHORS				5d PRC	611102		
0. AUTHORS Srinivasan Chandrasek	ar Kevin Trumble M	lart Ffa		Ju. TKC	KOJECI NUMBER		
Srinivasan Chandrasekar, Kevin Trumble, Mert Efe				5e. TAS	5e. TASK NUMBER		
				5f. WO	5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAMES AND ADDRESSES Purdue University 155 South Grant Street					8. PE NUMI	RFORMING ORGANIZATION REPORT BER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES)					10. SP AR(	PONSOR/MONITOR'S ACRONYM(S) D	
U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211					11. SPONSOR/MONITOR'S REPORT NUMBER(S) 60789-MS.21		
12 DISTRIBUTION AVAILIBILITY STATEMENT							
Approved for Public Rel	ease; Distribution Un	limited					
13. SUPPLEMENTAR The views, opinions and of the Army position, po	Y NOTES //or findings contained olicy or decision, unle	l in this report are those ss so designated by othe	e of the er docu	e author(s) and umentation.	l shoul	ld not contrued as an official Department	
14. ABSTRACT A study has been ma microstructure and t system. Two method wedge sliding. Cont novel shear textures.	ade of the interacti exture in structura ls are used to import rolled deformation , i.e., with basal pl	ve effects of large-s l hexagonal close p ose controlled defor paths accessed by anes tilted to the sh	strain acked matic LSEN eet su	deformation l metals, without a large st M have enablished a large st urface, and	n and th ma rain e oles c fine/u	deformation paths on Ignesium as the model material extrusion machining (LSEM) and reation of Mg AZ31B sheet with ltrafine grained microstructures. Field measured using particle image	
15. SUBJECT TERMS metals, deformation pro	becessing, machining, u	Iltrafinegrained microst	tructure	e, severe plas	ic defo	ormation, texture	
16. SECURITY CLASS a REPORT ID ARSTR	SIFICATION OF:	17. LIMITATION ABSTRACT	OF	15. NUMBE OF PAGES	R 19a Sri	a. NAME OF RESPONSIBLE PERSON inivasan Chandrasekar	
		UU			19t 76	b. TELEPHONE NUMBER 55-494-3623	

Т

Γ

٦

# **Report Title**

Final Report: Research Area 10: Synthesis and Processing of Materials: Enhancing Deformation Processing Capability of Structural HCP Metals

# ABSTRACT

A study has been made of the interactive effects of large-strain deformation and deformation paths on microstructure and texture in structural hexagonal close packed metals, with magnesium as the model material system. Two methods are used to impose controlled deformation - large strain extrusion machining (LSEM) and wedge sliding. Controlled deformation paths accessed by LSEM have enables creation of Mg AZ31B sheet with novel shear textures, i.e., with basal planes tilted to the sheet surface, and fine/ultrafine grained microstructures. The development of the shear texture has been correlated with the deformation field measured using particle image velocimetry (PIV). The shear texture, in combination with a fine grain size, has been shown result in enhanced combinations of strength and formability for LSEM sheet, compared to conventional rolled sheet. Furthermore, continuous sheet forms can be created in a single deformation step by LSEM by exploiting the adiabatic confinement of shear deformation intrinsic to the process, thereby minimizing the need for external heating. Under wedge sliding deformation, a new fluid-like flow and plastic instability has been discovered. This has also been identified as a key mechanism for particle formation and delamination wear in sliding, besides providing insights into how vortex-like flows develop and their relation to microstructure characteristics in ductile metals.

# Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received	Paper
03/03/2015	14 Koushik Viswanathan, Anirban Mahato, Srinivasan Chandrasekar. Nucleation and propagation of solitary Schallamach waves, Physical Review E, (01 2015): 0. doi: 10.1103/PhysRevE.91.012408
03/03/2015	17 D. Sagapuram, Z. Wang, C. Saldana. Thermal stability of nanotwinned and nanocrystalline microstructures produced by cryogenic shear deformation, Philosophical Magazine, (10 2014): 1. doi: 10.1080/14786435.2014.958590
08/27/2014	8 Dinakar Sagapuram, Mert Efe, Kevin Trumble, Srinivasan Chandrasekar. Enabling shear textures and fine-grained structures in Magnesium sheet by machining-based deformation processing, IOP Conference Series: Materials Science and Engineering, (08 2014): 12155. doi:
08/27/2014	7 Anirban Mahato, Yang Guo, Narayan Sundaram, Srinivasan Chandrasekar. Surface folding in metals: a mechanism for delamination wear in sliding, Proceedings of the Royal Society A: Mathematical, Physical and Engineering Sciences, (07 2014): 20140297. doi:
08/27/2014	9 Anirban Mahato, Yang Guo, Ho Yeung, Srinivasan Chandrasekar. Surface flow in severe plastic deformation of metals by sliding, IOP Conference Series: Materials Science and Engineering, (08 2014): 12016. doi:
08/27/2014	11 Anirban Mahato, Yang Guo, Narayan Sundaram, Tejas Murthy, Christopher Saldana, Srinivasan Chandrasekar. Unconstrained plastic flow at surfaces in sliding and cutting, International Journal of Precision Technology, (12 2013): 370. doi:
_	

TOTAL: 6



# (d) Manuscripts

Received		Paper
08/22/2012	1.00	Narayan Sundaram, Yang Guo, Srinivasan Chandrasekar. Mesoscale Folding, Instability, and Disruption of Laminar Flow in Metal Surfaces, Physical Review Letters (in press) (06 2012)
TOTAL:		1
Number of M	Ianus	cripts:
		Books
<u>Received</u>		Book
TOTAL:		
<u>Received</u>		Book Chapter
03/03/2015 1	16.00	Yang Guo, Narayan Sundaram, Srinivasan Chandrasekar, Airban Mahato. In Situ Study of Plastic Flow at Sliding Metal Surfaces, Cham (ZG) Switzerland: Springer International Publishing, (01 2015)
08/27/2014 1	10.00	Dinakar Sagapuram, Mert Efe, Wilfredo Moscoso, Srinivasan Chandrasekar, Kevin Trumble. Non-Basal Textures in Magnesium Alloy Strips produced by Extrusion Machining, Hoboken, New Jersey: Wiley-TMS, (03 2014)
TOTAL:		2

# **Patents Submitted**

## Awards

Andrew B. Kustas - Bilsland Dissertation Fellowship, Purdue University Koushik Viswanathan - Bilsland Dissertation Fellowship, Purdue University

	Graduate Stud	ents			
NAME	PERCENT_SUPPORTED	Discipline			
Kevin Chaput	0.10				
Andrew Kustas	0.10				
FTE Equivalent:	0.20				
Total Number:	2				
Names of Post Doctorates					
NAME	PERCENT SUPPORTED				
Dinakar Sagapuram	0.05				
Ho Yeung	0.10				
FTE Equivalent:	0.15				
Total Number:	2				
	Names of Faculty S	upported			
NAME	PERCENT_SUPPORTED	National Academy Member			
Srinivasan Chandrasekar	0.00				
Kevin Trumble	0.02				
W Dale Compton	0.00	Yes			
FTE Equivalent:	0.02				
Total Number:	3				
Names of Under Graduate students supported					
NAME	PERCENT_SUPPORTED				

FTE Equivalent: Total Number:

Student Metrics This section only applies to graduating undergraduates supported by this agreement in this reporting period
The number of undergraduates funded by this agreement who graduated during this period: 0.00 The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields: 0.00
The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields: 0.00
Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale): 0.00 Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering: 0.00
The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00
The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

### Names of Personnel receiving masters degrees

NAME

**Total Number:** 

## Names of personnel receiving PHDs

<u>NAME</u>

**Total Number:** 

## Names of other research staff

NAME

PERCENT\_SUPPORTED

FTE Equivalent: Total Number:

## Sub Contractors (DD882)

**Inventions (DD882)** 

**Scientific Progress** 

See Attachment

## **Technology Transfer**

We are closely interacting with M4 Sciences LLC - a Purdue based start-up that has commercialized research from our group; and Seco Tools, part of Sandvik (sweden)- in exploring development of machining-based deformation processes for sheet and rod making from structural metals, as well as for control of shear bands in component machining. M4 is a licensee of our patents and the current provisional application is under discussion with both Seco and M4.

Other interactions with Saint Gobain, MA, and Diamond Innovations, OH

#### Introduction

Light-weight alloys of hexagonal close-packed (HCP) metals, e.g., Mg, Ti, in fine-grained sheet forms hold extraordinary promise for structural applications in the DoD sectors. Production of metal sheet intrinsically requires very large total reduction strains. Currently, these large strains are imposed incrementally, in many-stage rolling processes, usually starting from large cast ingots and slabs. The as-cast ingots or slabs typically undergo breakdown rolling or forging for homogenization followed by multiple hot-rolling stages to accomplish the necessary large reductions. Oxidation in hot-rolling typically limits surface quality, requiring surface conditioning (e.g., pickling, scalping) prior to the final cold reduction. Cold-rolling is also conducted in multiple steps, and for HCP alloys often requires as many intermediate annealing treatments in order to avoid fracture. The multi-stage processing involves large dissipations due to friction at the die-material interfaces and the energy consumed by the annealing steps. Deformation processing of Mg and Ti alloys to sheet forms, with fine-grained microstructures (<10 micrometre) of high strength and formability, is constrained by the poor workability of these alloys, a consequence of the limited number of slip systems operative in these HCP metals at near-room temperature. Additional constraints to the deformation processing arise from the propensity of these alloys to segment, crack and/or shear band at the conditions typical for realizing fine-grained microstructures. Moreover, the resulting crystallographic textures typically have very strong components of basal plane alignment, limiting sheet formability for the end user. These problems are particularly acute in the case of Mg alloys, wherein the enabling deformation processing technology to achieve

#### Problem/Objective

The proposed work seeks to advance the state-of-the-art of deformation processing science of HCP metals through a fundamental study of how unique, interactive combinations of deformation conditions, viz., hydrostatic pressure, strain rate, temperature and strain, can be used to effect microstructure refinement and novel crystallographic textures, while minimizing segmentation and shear banding.

sheet forms having fine-grained microstructures and high formability on a commercial scale is currently lacking.

#### Approach

The frameworks for effecting wide-ranging combinations of deformation conditions are 1) a hybrid cutting-extrusion process – Large Strain Extrusion Machining (LSEM) [1-3] – which can impose controlled combinations of strain and strain rate in the chip; and 2) wedge sliding which can impose very large strains on surfaces through repeated application of sliding passes. The nature of the flow can be varied by controlling the wedge rake angle (incidence angle).

Large Strain Extrusion Machining (LSEM) - In contrast to conventional machining, wherein the chip (sheet) "peeled away" from the workpiece material is unconstrained, a constraining edge is applied to the chip at the point of its formation in LSEM. Thus, 'extrusion' occurs simultaneously with cutting in a process of constrained chip formation, the constraint converting the chip formation into a bulk deformation processing operation. Depending on the type of constraint imposed, the chip material emerges from the deformation zone as sheet, foil or rod. In the present study, plane strain LSEM wherein the chip is created as a foil/sheet is the configuration of primary interest.

The deformation conditions of strain, strain rate and temperature can be varied systematically by changing the extent of the constraint and the machining speed. Equally importantly, controlled shear deformation can be applied and the deformation path can be controlled, enabling creation of materials with different deformation states (e.g., texture, microstructure). Besides its important capabilities for studying deformation of metal alloys the process has significant potential for large-scale processing of alloys of poor workability into sheet/foil form.

*Wedge Sliding* - This framework imposes controlled combinations of strain and strain rate in thick surface layers of a workpiece by the sliding action of a hard wedge against the workpiece surface [4]. The incidence angle of the wedge with respect to the workpiece surface is small thereby, suppressing chip formation and material removal. The deformation conditions imposed on the workpiece surface are controlled by the wedge incidence angle, the interaction depth and the velocity of sliding. Very large strains can be effected by use of multiple sliding passes. This wedge sliding models the unit interaction in a variety of surface deformation processes such as burnishing, surface mechanical attrition, friction stir processing and peening. In addition, it is a model system for understanding sliding wear due to abrasion and adhesion, since the wedge is representative of a single asperity on a surface. Details of this wedge sliding may be found in Ref. [4].

*Measurements* - The two experimental frameworks, use in the studies to date, are fully instrumented to measure input parameters such as interaction depth, forces, sliding/cutting velocity and constrain parameters. Deformation parameters of strain and strain rate in the deformation zone are characterized in situ using novel high speed imaging techniques, and particle image velocimetry (PIV). Additional capability for measuring the temperature in the deformation zone using infra-red thermography is currently being set up. The acquisition of infra-red thermographic camera system has been facilitated by the DURIP grant awarded in Month 2013. Complementing the deformation measurements is characterization of the texture and microstructure of the deformed material using transmission

electron microscopy (TEM), EBSD, X-ray methods and optical metallography. Mechanical properties and formability of the deformed material are studied using hardness, tensile and biaxial tensile test (limiting dome height (LDH)) methods. Formability testing by LDH will be especially valuable for understanding fundamental deformation characteristics of shear-textured sheet and comparison with rolled sheet.

*Modeling* - The deformation characteristics are being analyzed using various continuum approaches structured around finite element analysis, crystal plasticity and texture simulation.

#### **Principal results**

The work has focused on developing the experimental framework for imposing controlled large-strain deformation for creating fine-grained metallic sheet forms and surfaces. This included *in situ* deformation characterization to effect various interactive combinations of the deformation; characterization of texture and microstructure in sheet created by shear-based deformation processing (via LSEM), including correlations of the deformation (path, magnitude) with texture and microstructure; control and suppression of segmentation/localized plastic flow in difficult-to-work materials such as Mg and Ti alloys; mapping of deformation conditions (strain, strain rate, temperature) pertaining to homogeneous and localized flow types; and fundamental studies of plastic flow and material removal at the meso scale in wedge sliding. The principal findings are summarized below:

- 1) High-speed imaging, in conjunction with the particle image velocimetry (PIV) based image correlation, has been implemented successfully to characterize the phenomenological features of material flow and quantify deformation zone parameters (strain, strain rate, and strain path tensorial quantities) at high spatial resolution (~1 µm) in LSEM, wedge sliding, and unconstrained cutting configurations. A key advance has been its application to map the transient (non-steady) deformation fields under conditions intermediate to those of sliding (no material removal) and cutting [5]. The material pile up ahead of the tool associated with this transient flow has been identified to be critical in triggering the inhomogeneous deformation and pattern formation in the deformed material (chip/surface). Furthermore, PIV has been also applied to quantify parameters corresponding to the shear-banded flow in highly cold-worked metals, representative of ultrafine-grained (UFG) and nanostructured materials processed using top-down SPD approaches.
- 2) The deformation capacity of HCP metal systems is mainly limited by the plastic flow localization that acts as a precursor to fracture. For example, in Mg alloys, the non-availability of sufficient slip systems at near-ambient temperatures contributes to flow localization and segmentation, whereas in Ti alloys, the low thermal diffusivity is believed to result in thermal (flow-stress) softening and flow localization. In conventional machining chip formation, the unconstrained flow in the deformation zone that enables stack-of-cards like slip and reduces the load bearing capacity of the chip [6], and material pile-up (prow formation) ahead of the tool [5] have been found to further contribute to flow localization. However, by constraining the back side of the chip/sheet using a constraint placed directly across the cutting tool, the propensity for stack-of-cards like slip and of prow formation can be minimized or eliminated. We have demonstrated the suppression of flow localization in Mg and Ti alloys (CP Ti and Ti-6AI-4V) using a sufficient constraint magnitude in LSEM [5, 7]. This suppression of flow localization using LSEM has important implications not only for the enhanced deformation processing of sheet metal with uniform microstructures, but also for machining of advanced structural alloys (Ti-6AI-4V, Ni-based superalloys), where localized flow (e.g., shear banding) has adverse consequences for tool life and machined surface quality.
- 3) Constrained chip (sheet) formation by large strain extrusion machining (LSEM) has been used to create Mg alloy AZ31B sheet forms having homogeneous microstructures with controllable grain sizes (200 nm-6um) and shear textures. The shear textures, characterized by the basal planes inclined away from the sheet surface, are quite different from those prevalent in rolled sheet (basal texture). The variation of basal plane inclination (in the 32-53° range) through strain path control by adjusting the constraint level has been demonstrated [3]. Enhancements in tensile strength and ductility have been shown for LSEM sheet by controlling the grain size and texture. Improvements in formability under biaxial tension have been also assessed using the limiting dome height (LDH) tests, wherein a 50% increase in LDH has been observed for the LSEM sheet over conventional rolled sheet [7]. Further in situ analyses have provided quantification of the spatial-temporal evolution of strain and strain rate fields under tensile deformation of sheet specimens. The tensile flow characteristics of Mg sheet have been found to be radically different from those of more conventional cubic metals (Cu, Al), in terms of both necking and fracture. The material flow, flow localization including (diffuse and local) necking and shear banding, and fracture were highly dependent on the sheet texture. Basal textures that constrain basal slip resulted in early flow localization. In contrast, large levels of uniform strain (above 0.2) nearly up to the point of fracture were realized in the shear-textured specimens favorably oriented for basal slip. Enhanced combinations of strength and formability due to grain size reduction and shear-texturing will have important implications for practical application of Mg sheet, and also for LSEM as a potential commercial deformation process. More recently, processing of high-performance Ti-

6AI-4V alloy strips with UFG microstructure (~100 nm grain size) has been demonstrated through the implementation of carbide tools in an LSEM configuration.

4) Using high-resolution, *in situ* imaging of a hard, wedge-shaped model asperity sliding against a metal surface, we have demonstrated a new mechanism for particle formation and delamination wear [4, 8]. Damage to the residual surface is caused by the occurrence of folds on the free surface of the prow-shaped region ahead of the wedge. This damage manifests itself as shallow crack-like features and surface tears, which are inclined at very acute angles to the surface. The transformation of folds into cracks, tears and particles is directly captured. Notably, a single sliding pass is sufficient to damage the surface, and subsequent passes result in the generation of platelet-like wear particles. Tracking the folding process at every stage from surface bumps to folds to cracks/tears/particles ensures that there is no ambiguity in capturing the mechanism of wear. Because fold formation and consequent delamination are quite general, our findings have broad applicability beyond wear itself, including implications for design of surface generation and conditioning processes.

#### Future work

A detailed study of the flow instabilities in machining and machining-based deformation processing is envisaged, driven by considerations of suppression of instabilities in deformation processing, machining and sliding wear. With regards to suppression of flow instabilities, approaches based on deformation zone geometry design and initial material microstructure/texture will be explored. The role of temperature in influencing the flow will be studied using infrared (IR) thermography of the deformation zone. This will utilize an IR camera recently acquired under the auspices of an Army DURIP award. Lastly, the possible estimation of flow stress behaviour of metals at large strain and high strain rates using machining tests will be assessed.

#### References

1. M. Efe, W. Moscoso, K.P. Trumble, W.D. Compton and S. Chandrasekar, Mechanics of Large Strain Extrusion Machining and Application to Deformation Processing of Magnesium Alloys, Acta Materialia, 60, 2031-42, 2012.

2. Y. Guo, M. Efe, W. Moscoso, D. Sagapuram, K.P. Trumble, and S. Chandrasekar, Deformation Field in Large Strain Extrusion Machining and Implications for Deformation Processing, Scripta Materialia, 66-5, 235-238, 2011

3. D. Sagapuram, M. Efe, W. Moscoso, S. Chandrasekar and K.P. Trumble, Controlling texture in magnesium alloy sheet by shear-based deformation processing, Acta Materialia, 61, 6843-56, 2013.

4. N. Sundaram, Y. Guo, and S. Chandrasekar, Mesoscale Folding, Instability, and Disruption of Laminar Flow in Metal Surfaces, Physical Review Letters, 109, 106001, 2012.

5. D. Sagapuram, H. Yeung, Y. Guo, A. Mahato, R. M'Saoubi, W.D. Compton, K.P. Trumble and S. Chandrasekar, On flow instabilities in cutting of metals and their control, CIRP Annals Manufacturing Technology, 2015 (accepted)

6. D. Sagapuram, R. M'Saoubi, K.P. Trumble, S. Chandrasekar. Shear Band Strain, Width and Microstructure in High-Velocity Shear Deformation. Abstract: S-03-705, 17<sup>th</sup> U.S. National Congress on Theoretical and Applied Mechanics, Michigan State University, June 15-20, 2014.

7. D. Sagapuram, A.B. Kustas, W.D. Compton, K.P. Trumble and S. Chandrasekar, Direct single-stage processing of lightweight alloys into sheet by hybrid cutting-extrusion, ASME Journal of Manufacturing Science and Engineering, 2015 (under review).

8. A. Mahato, Y. Guo, N. Sundaram and S. Chandrasekar. Surface Folding in Metals: A Mechanism for Delamination Wear in Sliding. Proceedings of the Royal Society A 470, 20140297, 2014.