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Final Report: Nanocrystalline MAX/Mg Composites with Exceptional Properties

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9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES)
U.S. Army Research Office
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The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT
Understanding the microstructure and mechanical properties of Mg-MAX composites
Composites were fabricated by pressureless melt infiltration of Mg and Mg alloys into TiC and Ti2AlC porous preforms. Ti2AlC is a layered machinable ternary carbide (MAX), which is relatively light and stiff. Pure Mg and three Al-containing Mg alloys, AZ31, AZ61 and AZ91 were used as matrices. When the matrix Al content was ? 6 wt.%, the best mechanical properties were achieved for all the composites fabricated. We also studied the effect of

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Mg/MAX composites, mechanical properties, damping, ripplocations

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19a. NAME OF RESPONSIBLE PERSON
Michel Barsoum

19b. TELEPHONE NUMBER
215-895-2338

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Report Title

Final Report: Nanocrystalline MAX/Mg Composites with Exceptional Properties

ABSTRACT

Understanding the microstructure and mechanical properties of Mg-MAX composites
Composites were fabricated by pressureless melt infiltration of Mg and Mg alloys into TiC and Ti2AlC porous preforms. Ti2AlC is a layered machinable ternary carbide (MAX), which is relatively light and stiff. Pure Mg and three Al-containing Mg alloys, AZ31, AZ61 and AZ91 were used as matrices. When the matrix Al content was ? 6 wt.%, the best mechanical properties were achieved for all the composites fabricated. We also studied the effect of reinforcement particle size. For fine-grained reinforcements - 50 vol.% TiC-AZ61 - the elastic modulus was 175±5 GPa, Vickers hardness was 3.4±0.3 GPa, and the ultimate compressive strengths was 1028±5 MPa. The enhancements in elastic and mechanical properties are attributed to finer grained Mg-matrices, the presence of Al in the matrices which enhances the wetting TiC and Ti2AlC by Mg to create strong interfaces and finer reinforcement particle sizes, that lead to a better mechanical interlocking. In addition, due to Ti2AlC inherent mechanical energy dissipation, damping properties of Mg-Ti2AlC composites were measured to be higher than their TiC reinforcement counterparts. Quite recently we discovered a new micromechanism in the deformation of layered solids; bulk ripplocations, whose existence will upend much of our understanding of the deformation of layered solids.

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
01/04/2017	23	Matthais Agne and M. W. Barsoum. Enthalpy of Formation and Thermodynamic Parameters of the MAX Phase, V2AlC., J. Alloy Compds., ( ): 218. doi:
10/01/2014	14	Babak Anasori, El'ad N. Caspi, Michel W. Barsoum. Fabrication and mechanical properties of pressureless melt infiltrated magnesium alloy composites reinforced with TiC and Ti2AlC particles, Materials Science and Engineering: A, (11 2014): 0. doi: 10.1016/j.msea.2014.09.039
10/23/2015	16	M. Shamma, , E. Caspi, , B. Anasori, , B. Clausen, , D. Brown, , S. Vogel, , V. Presser, , S. Amini, , O. Yeheskel, , M.W. Barsoum. In situ Neutron Diffraction Eviden For for Fully Reversible Dislocation Motion in Highly Textured Polycrystalline Ti2AlC Samples, Acta Materiala, (08 2015): 51. doi:
10/28/2015	17	Matthias T. Agne, Babak Anasori, Michel W. Barsoum. Reactions Between Ti2AlC, B4C, and Al and Phase Equilibria at 1000 °C in the Al-Ti-B-C Quaternary System, J. Phase Equilibria and Diffusion, (02 2015): 0. doi: 10.1007/s11669-015-0371-9
11/05/2015	19	B. Anasori, M. W. Barsoum. Reversible dislocation motion and microcracking in plastically anisotropicsolids under cyclic spherical nanoindentation, MRS Communications, (08 2013): 245. doi:
12/16/2016	18	M. T. Agne, M. Radovic, M. W. Barsoum. Stability of V2AlC with Al in the 800 to 1000 °C Temperature Range and in situ Synthesis of V2AlC/Al Composites, J. of Alloys and Compounds, (08 2015): 279. doi:
12/16/2016	20	Babak Anasori and Michel W. Barsoum. Energy Damping in Magnesium Alloy Composites Reinforced with TiC or Ti2AlC Particles, Materials Science and Engineering A, (08 2015): 53. doi:
12/16/2016	21	Jacob Gruber, Andrew C. Lang, Justin Griggs, Mitra L. Taheri, Garritt J. Tucker, Michel W. Barsoum. Evidence for Bulk Ripplocations in Layered Solids, Scientific Reports, ( ): 33451. doi:
12/21/2016	22	J. Gruber, A. Lange, J. Griggs, M. Taheri, G. Tucker and M. W. Barsoum. Evidence for Bulk Ripplocations in Layered Solids, Sci. Rep., ( ): 33451. doi:

TOTAL: 9

Number of Papers published in peer-reviewed journals:

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

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Invited Talks (M. W. Barsoum)
Plenary Lecture: 10th Inter. Conf. on High Perform. Ceram.; Nanchang; China, Nov. 2017.

Plenary Lecture: Long-Period Stacking Ordered Structure, Kyoto, Dec., 2016 Japan.

North Carolina State Univ., Chapel Hill, NC; Nov. 2016.

Texas A&M, College Station, TX, Sept. 2016.

MINATEC, Institute Polytechnique de Grenoble, Grenoble, France, March 2016.

Cambridge University, Cambridge, UK, Oct. 2015.

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received   Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received   Paper

TOTAL:

Number of Manuscripts:

Books

Received   Book

08/28/2012 1.00 Babak Anasori, Michel W. Barsoum. ON THE EFFECT OF Ti2AlC ON THE FORMATION OF THERMALLY STABLE Mg NANO GRAINS, Hoboken, NJ, USA: John Wiley & Sons, Inc., (03 2012)

08/28/2012 4.00 Babak Anasori , Shahram Amini, Volker Presser , Michel W. Barsoum. NANOCRYSTALLINE Mg-MATRIX COMPOSITES WITH ULTRAHIGH DAMPING PROPERTIES, Hoboken, NJ, USA: John Wiley & Sons, Inc., (02 2011)

TOTAL: **2**

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

M. W. Barsoum
Foreign Member of the Royal Swedish Academy of Engineering Sciences, 2016.

Chair of Excellence, Nanosciences Found., Univ. Foundation Grenbole Alpes, France, 2016.

Elsevier Scopus, List of 300 most Cited Authors in Materials Science and Engineering, 2016.

ASM Delaware Valley Materials Person of the Year, 2016.

Visiting Professor, Grenoble Instit. of Tech. Grenoble, France; Winter, 2016.

Leverhulme Trust Visiting Professorship, Imperial College, London, UK; Fall 2015.

Graduate Students

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

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

NAME
Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Technology Transfer

We worked with Ray Decker from Thixomat for a few months to try and find applications for the composites. We sent them samples and they tested them. An application did not materlize however.

REPORT DOCUMENTATION PAGE

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1. AGENCY USE ONLY (Leave Blank)

2. REPORT DATE 08/31/2014

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Nanocrystalline MAX/Mg Composites with Exceptional Properties

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6. AUTHOR(S)
M. W. Barsoum

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Drexel University, Philadelphia PA 19104

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12 a. DISTRIBUTION / AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

12 b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)
Understanding the thermodynamic stability of Al-MAX composites

Composites were fabricated by pressureless melt infiltration of Mg and Mg alloys into TiC and Ti_2AlC porous preforms. Ti_2AlC is a layered machinable ternary carbide (MAX), which is relatively light and stiff. Pure Mg and three Al-containing Mg alloys, AZ31, AZ61 and AZ91 were used as matrices. When the matrix Al content was ? 6 wt.%, the best mechanical properties were achieved for all the composites fabricated. We also studied the effect of reinforcement particle size. For fine-grained reinforcements - 50 vol.% TiC-AZ61 - the elastic modulus was 175±5 GPa, Vickers hardness was 3.4±0.3 GPa, and the ultimate compressive strengths was 1028±5 MPa. The enhancements in elastic and mechanical properties are attributed to finer grained Mg-matrices, the presence of Al in the matrices which enhances the wetting TiC and Ti2AlC by Mg to create strong interfaces and finer reinforcement particle sizes, that lead to a better mechanical interlocking. In addition, due to Ti_2AlC inherent mechanical energy dissipation, damping properties of Mg-Ti_2AlC composites were measured to be higher than their TiC reinforcement counterparts. Recently we also discovered a new micromechanism in the deformation of layered solids; bulk ripplocations, whose existence will upend much of our understanding of the deformation of layered solids.

14. SUBJECT TERMS

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REPORT DOCUMENTATION PAGE (SF298)
(Continuation Sheet)

Final Report

Submitted to: Dr. David M. Stepp
U.S. Army Research Office
AMSRL-RO-PM (Materials Science Division)

Title: Nanocrystalline MAX/Mg Composites with Exceptional Properties

Statement of the Problem Studied

The original aim of this work was to fabricate and test Mg-MAX composites and characterize and understand their mechanical and extraordinary damping properties. More recently, we sought to do the same for Al-MAX composites. Another longstanding goal was to understand the origins of damping observed in the MAX and other layered solids and in hexagonal metals. As discussed below, both aims have been for the most part accomplished.

Summary of Our Most Notable Accomplishments

During the course of this proposal 16 refereed papers were published and several others are planned. The papers published were on various aspects of Mg-MAX composites and our continued efforts to understand the kinking non-linear elasticity, which is at the origin of the damping properties measured in the composites we made. This continued and persistent approach led us to – as discussed below – a major discovery concerning a new micromechanism in the deformation of layered solids: bulk ripplocations.

A summary of our major accomplishments - with more emphasis on our new findings - are:

a) We fabricated and characterized Mg/MAX composites by pressureless melt infiltration of Mg and Mg alloys into porous preforms of TiC and Ti_2AlC, a member of the MAX phases. In this study, pure Mg and three commercially available, aluminum-containing Mg alloys, AZ31, AZ61 and AZ91 were used as matrices. When the matrix Al content is ≥ 6 wt.%, the best mechanical properties were achieved for all the composites fabricated herein. We also studied the effect of reinforcement particle size by using two different particle sizes. In the case of fine particle- 50 vol.% TiC-AZ61, the elastic modulus was 175±5 GPa, Vickers hardness was 3.4±0.3 GPa, and the ultimate compressive strengths was 1028±5 MPa. The enhancements in elastic and mechanical properties were attributed to finer grained Mg-matrices, the presence of Al in the matrices which enhances the wetting TiC and Ti_2AlC by Mg to create a strong interface and finer reinforcement particle sizes, which the latter two lead to a better mechanical interlocking. In addition, due to the inherent mechanical energy dissipation of Ti_2AlC, the damping properties of the Mg-Ti_2AlC composites measured were higher than their TiC reinforcement counterparts.[1,2]
b) We also fabricated and characterized Ti_3SiC_2 and Cr_2AlC MAX phase reinforced Mg and Mg-alloy metal matrix composites fabricated by pressureless melt infiltration. Pure Mg and Al-containing Mg-alloys (AZ31, AZ61 & AZ91), with varying Al content, were used a matrices, with loadings of ~ 45±1 vol. %. SEM revealed kinked, delaminated, and elongated

Ti_3SiC_2 grain, and non-deformed, rounded Cr_2AlC grain morphologies. Like in the Ti_2AlC/Mg composite system, increasing the Al wt. % content in the matrix enhanced the mechanical properties of the Ti_3SiC_2 composite system, but, somewhat surprisingly, had no effect on the properties of the Cr_2AlC composite system. At 1.9±0.1 GPa, 346±4 MPa and 617±10 MPa, the Vickers hardness, yield strength and ultimate compressive strengths, respectively, of the Ti_3SiC_2/AZ91 composite were the highest obtained. All composites fabricated exhibited fully and spontaneously reversible hysteresis loops. The mechanical properties were found to be sensitive to the reinforcement volume fraction. This work will be submitted for publication soon.

c) Given our success with reinforcing Mg-alloys with the MAX phases, the next question posed was can these results be reproduced with Al-matrices. To answer that question we tried to fabricate Al-MAX composites by pressureless MI. What we discovered was that pressureless MI cannot be used to fabricate Al matrix composites with Ti_2AlC, even when B_4C is incorporated in the melt to increase the infiltration kinetics. The main hurdle is that Ti_2AlC is *not* in equilibrium with Al at the processing temperatures required for infiltration and thus completely reacts with Al regardless of infiltration conditions. This work, led instead to a determination of the Ti-Al-B-C quaternary in the Al-rich corner. This was accomplished by fabricating two composites starting with Al, Ti_2AlC, and B_4C. The Ti_2AlC/B_4C powders were mixed in both 50/50 and 75/25 vol. % ratios and cold pressed into 53 % dense preforms. The preforms were pressureless MI in the 900 to 1050 °C temperature range with Al. Ten hour equilibration experiments were also conducted at 1000 °C. X-ray diffraction and scanning electron microscopy confirmed that neither Ti_2AlC nor B_4C was an equilibrium phase. A number of reaction phases – AlB_2, Al_3BC, TiB_2, TiC, $TiAl_3$ and Al_4C_3 – could be found in the non-equilibrated samples. However, the equilibrium phases were found to be Al, TiB_2, Al_3BC, and Al_4C_3 for the more B-rich composite and Al, TiB_2, TiC, and Al_4C_3 for the Ti-rich composite. From these results, the 1000 °C quaternary phase diagram adjacent to the Al-TiB_2-Al_4C_3 triangle and in the Al-rich corner was developed for the first time. This work is a requisite first step for the development and use of advanced composites in the Al-Ti-B-C system.[3]

d) In sharp contradistinction to Ti_2AlC and most MAX phases, V_2AlC is in equilibrium with molten Al, which prompted us to try and fabricate Al-V_2AlC composites. We were successful, but in the process of making these composites we realized that Al and V_2AlC were in equilibrium only at temperatures > 940 °C. Below that temperature the following reaction occurs: $3V_2AlC + 19\ Al \Rightarrow Al_4C_3 + 6Al_3V$. We used this information to determine the Al-rich corner of the V-Al-C phase diagram at 800 °C, for the first time. We also used that information to calculate the enthalpy of formation of V_2AlC and then used heat capacity results to derive the thermodynamic parameters of V_2AlC over the 300 to 1400 K temperature range, for the first time. Based on our work, the free energy of formation of V_2AlC was calculated to be ΔG_f (kJ/mol) = (-186 ±14) + 0.021 T.[4]

e) Returning to the composites. The nominal compositions were chosen so that *in situ* reaction would produce an Al-rich (75/25 vol. % Al/V_2AlC) and a V-rich (50/50 vol. % Al/V_2AlC) composites. To make these elementals powders were cold pressed to 200 MPa, heated to 1000 °C and reacted for 0.5, 2.5 or 10 h under flowing Ar. Water quenched samples produced two-phase Al-V_2AlC composites, but furnace cooled samples did not. Fully dense composites have not been realized yet and we thus do not have any mechanical properties.

However, the totality of these studies are needed if Al-V_2AlC and other advanced composites in the Al-V-C system are to be fabricated and tested. [5]

f) By far our most important discovery lately has been the identification of a fundamentally new deformation micromechanism in layered solids.[6] Layered materials are ubiquitous; they are present in geological formations, nuclear reactors, microcircuits, sensors, biomaterials and polymers - both natural and synthetic – and myriad others. They also are important players in the nanomaterials revolution. Consequently, their deformation has been studied extensively for decades, especially in geology, where many silicates are layered. It has also long been appreciated that kinking is a favored deformation mode in layered solids, such as mica, graphite, ice, the MAX phases (see below) among many others. For example, kink bands, KBs, have been observed in micas/biotites, at many scales, from the microscopic to the macroscopic.

Recently, a new defect termed a ripplocation (A. Kushima et al. Nano Letters **15**, 2015) – best described as an atomic scale ripple – was proposed to explain deformation in two-dimensional solids. In our work, we leveraged atomistic simulations of graphite to extend the ripplocation idea to bulk layered solids, and confirm that it is essentially a buckling phenomenon. In contrast to dislocations, bulk ripplocations have no Burgers vectors or polarities. In graphite, ripplocations are attracted to vacancies and other ripplocations, both within the same, and on adjacent layers, the latter resulting in kink boundaries. Furthermore, we obtained TEM evidence for bulk ripplocations in Ti_3SiC_2. The nucleation of delamination cracks, when atomic layers are loaded edge-on with, say, a spherical indenter, is an unambiguous signature of bulk ripplocations. [6]

We also directly imaged BRs in the TEM and conclusively showed that a c-strain component is associated with these defect. The latter is not possible if the defect was a basal dislocation. Furthermore, no g•b condition could be found that rendered these defects invisible again strongly suggesting they are *not* basal dislocations. [6]

Ripplocations are not only a fundamentally new deformation micromechanism, but are a topological imperative, since it is the only way atomic layers can glide relative to each other without breaking the all important in-plane bonds. The ramifications of their existence are profound, and will upend our current understanding of how graphite, layered silicates, the MAX phases, among many other plastically anisotropic solids deform. This work was published in Scientific Reports in 2016.[6]

There is little doubt at this time that the energy dissipated when the MAX phases are cyclically loaded is due to the nucleation and to and fro movement of bulk ripplocations. In this coming year, we will publish several papers showing experimental and other evidence for this important conclusion.

Interestingly enough, the origin of damping in the hexagonal metals is NOT ripplocations, but most probably the bowing out of dislocations in kink boundaries formed during the first loading cycle. This paper has been submitted for publication.

g) In 2015 we published a paper in Acta Mater. in which the abstract reads in part: "In this work careful analysis of in situ neutron diffraction patterns obtained, while cyclically loading highly textured polycrystalline of Ti_2AlC samples, provides compelling experimental evidence – in the form of fully reversible peak lattice elastic strain loops and peak widening and narrowing upon load cycling - for the existence of fully reversible dislocation motion. A comparison of the measured and calculated dislocation densities clearly shows that dislocation pileups alone cannot account for the experimental observations. Another micromechanism needs to be invoked. Based on the propensity of the MAX phases to deform by kinking, the micromechanism proposed is either the nucleation and annihilation of low angle kink boundaries, LAKB, and/or the bowing out of dislocations in the latter. This micromechanism plays a vital role during the initial deformation of layered and other plastically anisotropic solids such as hexagonal close-packed metals."[7]

Needless to add, in light of our discovery of ripplocations, these conclusions have to be modified and re-assessed in light of the existence of bulk ripplocations. Looking back at the results obtained however, we now see that neutron diffraction results were incompatible with basal dislocations and actually very strongly support the existence of BRs. A reappraisal of these results is ongoing and a paper will be submitted in the near future.

PhD Theses:

Babak Anasori, "Fabrication and Mechanical Properties of Magnesium Alloy Composites Reinforced with TiC and Ti_2AlC Particles", Materials Science and Engineering Department, Drexel University, 2014.

Justin Griggs, Investigation of the Reversible Hysteresis Effect in Hexagonal Metal Single Crystals and the MAX Phases. Drexel University, 2015.

MSc Thesis

Matthias Agne, "The Stability of Ti_2AlC and V_2AlC with Al, and the Synthesis of Composites in the Al-Ti-B-C and Al-V-C systems".

Patents Filed:

None.

References

1 Anasori, B. & Barsoum, M. W. Energy Damping in Magnesium Alloy Composites Reinforced with TiC or Ti_2AlC Particles. *Mater. Sci. Eng. A* **653**, 53–62 (2016).

2 Anasori, B., Caspi, E. & Barsoum, M. W. Fabrication and Mechanical Properties of Pressureless Melt Infiltrated Magnesium Alloy Composites Reinforced with Ti_2AlC and TiC Particles. *Mater. Sci. Eng. A* **618**, 511-522 (2014).

3 Agne, M. T., Anasori, B. & Barsoum, M. W. Reactions Between Ti_2AlC, B4C and Al and Phase Equilibria at 1000 °C in the Al-Ti-B-C Quaternary System. *J. Phase Equil. Diff.* **36**, 169–182 (2015).

4 Agne, M. T. & Barsoum, M. W. Enthalpy of Formation and Thermodynamic Parameters of the MAX Phase, V_2AlC. *J. Alloy Compds.* **665**, 218–224 (2016).

5 Agne, M. T., Radovic, M. & Barsoum, M. W. Stability of V_2AlC with Al in the 800 to 1000 °C Temperature Range and the in situ Synthesis of V2AlC/Al Composites. *J. Alloy Compds.* **666**, 279-286 (2016).

6 Gruber, J. *et al.* Evidence for Bulk Ripplocations in Layered Solids. *Sci. Rep.* **6**, 33451 (2016).

7 Shamma, M. *et al.* In situ Neutron Diffraction Evidence For for Fully Reversible Dislocation Motion in Highly Textured Polycrystalline Ti_2AlC Samples. *Acta Mater.* **98**, 51–63 (2015).