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LLNL-TR-738151

Manufacturing and Characterization of Ultra Pure Ferrous Alloys Final Report CRADA No. TC02069.0

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Disclaimer

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Manufacturing and Characterization of Ultra Pure Ferrous Alloys

Final Report
CRADA No. TC02069.0
Date Technical Work Ended: December 31, 2008

Date: June 2, 2009 Revision: 2

A. Parties

This project was a relationship between Lawrence Livermore National Laboratory (LLNL) and Caterpillar Inc.

Lawrence Livermore National Security, LLC
Lawrence Livermore National Laboratory
7000 East Avenue
Livermore, CA 94550
Donald Lesuer (LLNL retiree)
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Technical Center – Building A
14009 Old Galena Road
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Timothy E. McGreevy, Ph.D.
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B. Project Scope

This CRADA was a collaborative effort between the Lawrence Livermore National Security, LLC (formerly University of California)/Lawrence Livermore National Laboratory (LLNL) and Caterpillar Inc. (Caterpillar), to further advance levitation casting techniques (developed at the Central Research Institute for Materials (CRIM) in St. Petersburg, Russia) for use in manufacturing high purity metal alloys. This DOE Global Initiatives for Proliferation Prevention Program (IPP) project was to develop and demonstrate the levitation casting technology for producing ultra-pure alloys. The project was a collaborative effort between CRIM, Brown University, Caterpillar Inc. and LLNL to guide the levitation casting technology toward practical industrial applications. The goal of the pilot project was to determine in complete detail the effects of composition of high purity alloys on the microstructural development and thermo-mechanical properties of steels. The ability of the proposed approach to guide alloy and thermo-mechanical processing development was to be demonstrated.

The CRADA project anticipated the completion of the following major tasks and deliverables:

Project Year 1

Task 1.1: Develop melting technology under levitation conditions (2nd quarter) (CRIM/ Levitatsia-S LTD)
Task 1.2: Develop rolling thermal processing procedures (3rd quarter) (CRIM/ Levitatsia-S LTD)
Task 1.3: Develop numerical modeling to guide manufacturing of the test specimens and extraction of material properties from the raw experimental data (3rd quarter) (CAT and Brown)
Task 1.4: Characterize 30 compositions (4th quarter) (CRIM/ Levitatsia-S LTD, LLNL and Brown)

Project Year 2

Task 2.1: Characterize 45 compositions (4th quarter) (CRIM/ Levitatsia-S LTD, LLNL and Brown)
Task 2.2: Compute effect of processing parameters on microstructures and optimize processing (3rd quarter) (CRIM/ Levitatsia-S LTD, LLNL and Brown)

Project Year 3

Task 3.1: Characterize 49 compositions (2nd quarter) (CRIM/ Levitatsia-S LTD, LLNL and Brown)
Task 3.2: Perform numerical simulation to relate mechanical performance to measurable and controllable material properties (3rd quarter) (CAT and Brown)
Task 3.3: Publish project results in engineering journals (4th quarter) (CAT, CRIM/ Levitatsia-S LTD, Brown, and LLNL)

Project Year 4

Task 4: Apply project results to simulation of industrial material (4th quarter) (CAT)

Project Year 5

Task 5: Apply project results to simulation of industrial material (4th quarter)(CAT)
Task 6: Final Report (CAT and LLNL)

Deliverables:
The materials (high purity metal alloys) produced by CRIM/ Levitatsia-S LTD after casting and thermo-mechanical processing will be supplied to Caterpillar, Brown University and LLNL for their analysis. During the project approximately 124 different compositions will be cast and processed. It is expected that this as-processed material will be supplied at the end of the first quarter in years one and two. All other deliverables on the project will consist of quarterly progress reports. Periodic comprehensive reports that describe the results of all work completed will be made at the end of each year.

This CRADA was executed on August 16, 2004, and was originally designated as a five (5) year project. On November 21, 2005 a no-cost time extension request was executed, extending the

project for an additional fourteen (14) months, to October 16, 2010, due to a delay in the finalization of the contract with the Central Research Institute for Materials (CRIM) in Russia. The actual start date for this project was October 20, 2005. On March 27, 2006 an amendment was executed for this CRADA to designate Levitatsia-S LTD as the primary Russian Institute, and Central Research Institute of Materials (CRIM) as subcontractor to reflect the current structure of the partnership. All other terms and conditions remained the same.

The project was envisioned to be a three year project between Levitatsia-S Institute & CRIM, Brown University and Caterpillar. Year one lasted over three years and was only 30% completed due to lack of performance by the Russian Institute.

On November 25, 2008, LLNL received a notice of termination letter from Caterpillar, terminating their participation in the CRADA effective December 31, 2008. The Contract between LLNL and the Russian Institute was terminated on March 31, 2009.

C. Technical Accomplishments

Below is a table of deliverables LLNL received under this project, as well as the associated Russian Contract line item. No other deliverables were received from the Levitatsia Institute in Russia.

Item No.	Description of deliverable	Date Invoice Approved for payment
1	Part 1, Documentation Plan documenting completion of Task 1, 2, and 3	31 July 2006
8	Part 1, Institute Acquired Property Report	12 July 2006
9	Part 1, Subcontractor "Chrom", Ltd., St. Petersburg. Report	11August 2006
10	Part 1, Overhead	11 August 2006
11	Part 2, Task 1-Development of Project Plan and Purchase of Materials. Report describing research plan, Modification 01.	31 July 2006
13	Part 2, Task 2.2.1-Prepare Samples for Microstructural Analysis-1st Quarter. E-mail letter of work performed documenting completion of this task, Modification 01.	31 July 2006
17	Part 2, Task 2.3.1-Evaluate Microstructures-1st Quarter. E-mail letter of work performed documenting completion of this task, Modification 01.	31 July 2006
23	Part 2, Task 3.3.1-Perform Chemical Analysis-1st Quarter. E-mail letter of work performed documenting completion of this task, Modification 01.	31 July 2006
32	Part 2, Task 4.3.1-Ship 24 samples to LLNL and 24 Samples to Izhevsk-1st Quarter. Send e-mail shipment information 15 Days in advance to shipping the samples. The e-mail shall state the date shipment leaves "Levitatsia-S" and the date shipment leaves Russia and the date shipment is to arrive at US Customs. The e-mail shall also state the date shipment leaves "Levitatsia-S" and date shipment is to arrive at Izhevsk, Modification 01	31 July 2006
41	Part 2, Task 6.1.1-Heat Treat Forged Samples. E-mail letter of work performed documenting completion of this task, Modification 01.	31 July 2006
53	Part 2, Task 7.1-Evaluate Nitrogen and Oxygen Content. E-mail letter of work performed documenting completion of this task, Modification 01.	31 July 2006
57	Part 2, Task 8.1.1-Prepare Samples for Microstructure Analysis. E-mail	31 July 2006

Item No.	Description of deliverable	Date Invoice Approved for payment
	letter of work performed documenting completion of this task, Modification 01.	
61	Part 2, Task 8.2.1-Evaluate Microstructure of Deformed and Heat Treated Samples. E-mail letter of work performed documenting completion of this task, Modification 01.	31 July 2006
68	Part 2, Institute Acquired Materials and Property Report of property and materials actually purchased and compare it to the items listed in the attached Property Identification List. Any differences shall be clearly identified, Modification 01.	11 August 2006

Total spent to date = $72,720
Amount allocated = $245,000.00 under Foreign contract

According to LLNL, the first and only batch of payments for the above mentioned deliverables were sent to the institute on October 2006. Since that time there has been no further deliverables under the project and communication from Levistatsia started to decrease. There was no issue or concern with the communication between LLNL and Caterpillar.

While the Russians did not submit a "final report" for Year 1 to LLNL, they did complete work on six (6) compositions which included alloy samples sent to LLNL, as well as test data and technical reports on their findings. LLNL did some further analysis of the data. The samples were also sent to Caterpillar who also performed some analysis of them. The remaining material was then sent to Brown University who did some mechanical tests on the alloys.

From November 2006 to November 2008, Don Lesuer and Paris Althouse (the GIPP Program Manager) attempted no less than every three months to contact the Institute an dinquire on the status of the project and overdue deliverables. According to Dr. Lesuer and Ms. Althouse, Institute representatives would periodically respond with reasons as to why they had no deliverables.

Due to lack of performance from the Russian Institute, Paris Althouse notified the Institute and their representatives that unless deliverables were received by March 31, 2009, the contract for Year 1 would be descoped to reflect only those deliverables which had been received by LLNL. The remaining unspent foreign funds would be removed from the contract and returned to DOE (or redirected to other LLNL projects at DOE's discretion).

D. Expected Economic Impact

No positive economic impact was realized since the project was not completed.

D.1 Specific Benefits

Benefits to DOE
The project could have advanced the non-proliferation goals of the Initiatives for Proliferation program, a DOE program.

Benefits to Industry
The unique casting technology could have been used by Caterpillar material and process development studies. The US taxpayer could have benefited from the reduction of proliferation risks in Russia.

E. Partner Contribution

Caterpillar completed Task 1.3: "Develop numerical modeling to guide manufacturing of the test specimens and extraction of materials properties from the raw experimental data."

Additional Caterpillar deliverables were dependent upon tasks that were not completed by the Russian Team; as such, Caterpillar was not able to complete any other deliverables.

No subject inventions were created during the CRADA project.

F. Documents/Reference List

Reports

There were several reports based on the above limited work performed under this CRADA. The reports and data from the Russian Team were all forwarded to the Primary contact at Caterpillar (Leo Chuzhoy) and his associate (Patrick Zhao). The LLNL PI has since retired and the LLNL copies of these files remain in his custody.

Publications:

* Protected CRADA Information

- "Modeling of reaustenitization of hypoeutectoid steels with cellular automaton method", B.J. Yang, L. Chuzhoy, & M.L. Johnson, Computational Materials Science, Vol. 41, No. 2, pp. 186-194, May 2, 2007.*

- "Modeling of Microstructure Evolution of Athermal Transformation of Lath Martensite", Don H. Sherman, Bing J. Yang, Adrian V. Catalina, Ashwin A. Hattiangadi, Patrick Zhao,

Leo Chuzhoy and Micheal Johnson, Materials Science Forum, Vols. 539-543 (2007), pp. 4795-4800.

Copyright Activity

None

Subject Inventions

None

Background Intellectual Property

No Background Intellectual Property was disclosed by either party for this project.

G. Acknowledgement

Industrial Participant's signature of the final report indicates the following:

1) The Participant has reviewed the final report and concurs with the statements made therein.
2) The Participant agrees that any modifications or changes from the initial proposal were discussed and agreed to during the term of the project.
3) The Participant certifies that all reports either completed or in process are listed and all subject inventions and the associated intellectual property protection measures generated by his/her respective company and attributable to the project have been disclosed and included in Section E or are included on a list attached to this report.
4) The Participant certifies that if tangible personal property was exchanged during the agreement, all has either been returned to the initial custodian or transferred permanently.
5) The Participant certifies that proprietary information has been returned or destroyed by LLNL.

Timothy E. McGreevy, Program Manager, PD COE Advanced Materials — 7/28/2009 Date
Caterpillar Inc.

Donald Lesuer, LLNL Principal Investigator — 8/4/2009 Date
Paris Althouse, GIPP Program Manager
Lawrence Livermore National Laboratory

for Erik J. Stenehjem, Industrial Partnerships Director — 8/11/09 Date
Lawrence Livermore National Laboratory

Attachment I – Final Abstract

Manufacturing and Characterization of Ultra Pure Ferrous Alloys

Final Abstract (Attachment I)
CRADA No. TC02069.0
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B. Purpose and Description

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The CRADA project plan consisted of six major tasks and the following deliverables:

Deliverables:
The materials (high purity metal alloys) produced by CRIM/ Levitatsia-S LTD after casting and thermo-mechanical processing will be supplied to Caterpillar, Brown University and LLNL for their analysis. During the project approximately 124 different compositions will be cast and processed. It is expected that this as-processed material will be supplied at the end of the first quarter in years one and two. All other deliverables on the project will consist of quarterly progress reports. Periodic comprehensive reports that describe the results of all work completed will be made at the end of each year.

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C. Benefit to Industry

The unique casting technology could have been used by Caterpillar material and process development studies. The US taxpayer could have benefited from the reduction of proliferation risks in Russia.

D. Benefit to DOE/LLNL

The project could have advanced the non-proliferation goals of the Initiatives for Proliferation program, a DOE program.

E. Project Dates

Actual project dates were October 20, 2005 through December 31, 2008. Original project dates were designated as August 16, 2004 through August 16, 2009, and extended to October 16, 2010.