Solid-phase oxygen control system

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Project Title: Solid-Phase Oxygen Control System

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TRP Research Area: Transmutation Engineering
1. Introduction:

Liquid lead and lead-bismuth eutectic (LBE) are good candidates for the nuclear coolant in accelerator-driven systems or advanced reactors [1]. Corrosion of containment and structural materials presents a critical challenge in the use of these materials as a nuclear coolant in accelerator-driven systems or advanced reactors [2]. One way to reduce the corrosion in liquid lead and LBE and in the whole circuit is to protect the structural materials with a stable oxide layer, and that layer can be sustained, provided a certain level of oxygen concentration in the LBE. This sets a lower limit on the oxygen concentration in the LBE. An upper limit is set by the constraint that any solid oxides of Pb or Bi will not be formed in the liquid, which otherwise might contaminate the systems [1], or which otherwise might provide a source of oxygen that cannot be easily removed [3].

These effects have been confirmed by two separate experimental works performed in LANL (Task 13 under TRP) and in IPPE [3]: excess of oxygen may result in slagging in the circuit (i.e. disturbance of their thermal and hydraulic characteristics), while insufficient dissolved oxygen content may lead to dissociation of the oxide coating on the structural materials and corrosion processes.

As a result, for liquid lead or LBE to be used in any processes, it is vital that the oxygen content has to be carefully controlled. Active oxygen control has been determined as a key method to mitigate corrosion and coolant contamination in lead and lead-bismuth eutectic systems [4]. A straightforward technique for oxygen control system is the direct injection of oxygen/hydrogen or hydrogen/water gases mixed with an inert carrier gas (helium or argon). However, these gas-phase oxygen control schemes may lead to slag deposition and accumulation on component surface for long-term operations. Such slag deposition is very difficult to be cleaned up and results in complete failure of the entire system eventually [5].

To address aforementioned problems, we propose to adopt a solid-phase oxygen control scheme that can completely avoid the excessive slag deposition. In specific, we will design a solid mass exchanger filled with lead oxide (PbO spheroids). For the control optimization, inside the mass exchanger, the internal heater as well as one pump will be specially studied and designed.

When pumping oxygen-under-saturated coolant through a mass-exchanger, the lead oxides are dissolved in alloy and in this case a mass-exchanger is the generator of dissolved oxygen for coolant flow and loop. At the coolest leg of the loop, coolant will be easily under the condition of oxygen-saturated. While the coolant flow carries dissolved oxygen to the other heated legs, coolant is will become oxygen-under-saturated and no more contamination of coolant will be generated. By control coolant temperature in mass-exchanger, pumping rate, and pumping period, it is possible to control the dissolved oxygen supply to form the protective corrosion-resistant films on steel surface depending on monitored parameters from oxygen sensors.

We proposed to address the following questions: design the mechanical stable oxide medium with porosity; optimize the porous medium for passing and interacting with the liquid lead and lead bismuth; illustrate the flow characters in porous medium with surface reaction and the
exchange rate dependence on the flow rate and temperature, the oxygen controlling system design with filter for extra slag cleaning.

2. General Approaches and its Deficiencies:

A straightforward technique for oxygen control system is the direct injection of oxygen/hydrogen or hydrogen/water gases mixed with an inert carrier gas (helium or argon) (see Fig. 1). Another scheme is using oxygen-containing compounds with high oxygen pressure placed over the cover gas cavity of the circuit [6], as shown in Fig 2. However, in long-term operations, a large amount of slag deposition may be formed and accumulated on the component surfaces using this scheme, according to experience in LANL and IPPE operations (see Fig. 3). Such slag-deposition is very difficult to be cleaned up and can lead to a complete failure of the entire system eventually [5].

In gaseous oxygen supply, the formed oxides are dissolved partially, thus maintaining generation of dissolved oxygen and most of them are transferred and settled in different areas of the loop where their solution is hindered and non-controlled for different reasons. Thus, extra slag blockages are formed in a loop. Therefore, the gas phase methods do not solve completely the problem of continuous control of oxygen thermodynamic activity.

Usually, the eutectic components (lead and bismuth), lead and iron oxides form the basis of slag deposits. As an example, Table 1 presents the analysis results of some slag types [3]. The ratio of eutectic elements, lead and iron oxides in different slag deposits can vary significantly, however, their characteristic features are preserved. First of all, in spite of high mass fraction of eutectic elements, the slags represent, the non-transportable, non-pumped solid compound mixture, blocking different sections of loop. The lead oxide is found to be the primary binding material in slags. It is the lead oxide with an iron oxide impurity that provides for a solid porous space structure, the inner volume of which (cells) is filled with non-oxidized eutectic elements, i.e. lead and bismuth. This formation represents the combined-dispersed system, which is rather stable and indestructible, for example, by heating to 500 deg C, although the eutectic melting point is about 127 deg C.

<table>
<thead>
<tr>
<th>Facilities</th>
<th>Phase Element, mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PbO</td>
</tr>
<tr>
<td>VT-5 BEI, Obninsk</td>
<td>48.3</td>
</tr>
<tr>
<td>MOTsKTI, Moscow</td>
<td>45.5</td>
</tr>
<tr>
<td>OKBM, Nizhny Novgorod</td>
<td>~30</td>
</tr>
</tbody>
</table>

Table 1. Slag composition in loops.
Fig. 1. Gas-phase oxygen direct injection scheme.

Fig 2. Gas-phase oxygen control system
3. New Approach:

The solid-phase-method of control of oxygen TDA is the suggested method to use so as not to allow any negative side effect on the circuit. Rather it provides conditions for long-term operation corrosion resistance of materials in all operating modes of a reactor facility. This method also provides the possibility to implement the oxygen thermodynamic activity control using small-size devices which are highly reliable, simple to design and easy to operate. The dissolved oxygen generation in the solid-phase method and dissolved oxygen generation by gaseous oxygen supply into the loop are similar in physical and chemical nature.

In practices, slag still exists without gaseous oxygen supply (mixture of hydrogen, water vapor and inert gas) for long term operation. It is evident that the presence of slag can be explained by coolant interaction with oxygen or another oxidizer, for examples, water vapor. It is the coolant oxidation that is the starting reason for slag formation in a loop. The analysis of a considerable body of statistical data has shown that the primary source of oxygen ingress into a loop is its opening, especially, during maintenance operations.

In order to prevent the slag blockage of heat transfer surfaces, disturbances in pumps and valves operation, increase of hydraulic resistance of a loop, etc, the cleaning procedure before coolant pass through the mass-exchanger has to be considered. This clean procedure lies in injection gaseous mixture of hydrogen and inert gas into the coolant flow, as shown in Fig. 4. The bubbles of hydrogen containing gas are transferred with the coolant flow to slagging areas where solid lead oxides are reduced to lead which returns into the coolant composition.

Accordingly, hydrogen slag reduction is the process of coolant regeneration. The lead oxide reduction results in slag deposits destruction, in general, since the lead oxide is the main binding material of slags. So, not only the reduction product (lead), but also lead-bismuth (a part of the slag composition in the non-oxidized state before regeneration) returns into the coolant.
In regeneration procedure, in addition to the return of reaction products (lead and bismuth) into the coolant composition, the dust slag residues still exist in the coolant flow. The filters are suggested for cleaning the coolant flow from fine particles. Simultaneous hydrogen regeneration and coolant filtration are the most effective ways to prevent and to eliminate the critical situations in a loop associated with slag blockages.

Two main mechanisms of filtration process are realized: the mechanic trapping of impurity particles from alloy (Fig. 4) and the adhesion capture of impurities by the whole volume of filtering material. The filters are designed for a certain impurities capacity. In the process of impurity accumulation in a filter its cleaning efficiency drops, however, the coolant flow through the filter is not stopped. This peculiarity of developed filters is very important. The bubbles of hydrogen containing gasous mixture enter the filter with a coolant flow and reduce the lead oxides to lead and, in this way, clear the filter. As a result, the residues of only those impurities, which cannot be reduced by hydrogen, are concentrated in the filter. These are iron oxides, structural material particles, caused by material attrition, welding, and other impurities, which can be formed in a loop during its long-term operation.

The amount of irreducible slag residues is relatively low enabling the filter to be designed for the whole operation period of facilities without its replacement and removal from a loop. However, the reserved filter will still be considered in case of filter failure (Fig. 4).

![Diagram of the solid-phase oxygen controlling system.](Image)
This solid-phase method implies the use of solid-phase lead oxides (PbO spheroids) placed and retained in the limited-volume section of the circuit (reaction vessel of mass exchanger), as shown in Fig. 4. The oxygen is released and transported by the coolant flow through solid-phase lead oxides in contact with flowing liquid lead/alloy in the mass exchanger without extra slag generation. Hence, the use of mass-exchangers (generators of dissolved oxygen) is the effective technological procedure for slag prevention.

**Principle:**

The method of solid-phase oxygen control implies the use of solid-phase lead oxides placed and retained in the limited volume section of the circuit (reaction vessel of mass exchanger). In contact with following liquid lead/alloy, the PbO spheroids are dissolved releasing oxygen which is transported by the coolant flow through the circuit.

The principle concept of the process of dissolving the solid-phase oxide to maintain oxygen TDA follows from thermodynamic analysis of the system of the mixture of lead oxide and liquid lead/alloy. The kinetic characteristics of the process (or mass exchanger) however have to be investigated.

Accordingly, both dissolved oxygen content \( C_0 \) and its thermodynamic activity \( a_0 \) are controlled in lead-bismuth (lead). Relationship of these parameters is as follows [4]:

\[
a_0 = \frac{C_0}{C_{0S}},
\]

where \( C_{0S} \) is the oxygen saturation content in lead-bismuth (lead), and it is determined by the following formulae:

For liquid lead:

\[
\lg C_s = 3.44 - \frac{5240}{T}, \text{ wt. }%;
\]

and for Pb-Bi eutectic:

\[
\lg C_s = 1.18 - \frac{3400}{T}, \text{ wt. }%;
\]

Direction and intensity of the mass transfer processes in the eutectic and the circuit are determined by the absolute values of \( C_0 \) and \( a_0 \) and their variation. Taking into account continuity of these processes and their rather high mass transfer rate in some cases, it is expedient to perform permanent control of \( C_0 \) or \( a_0 \).

Current status of technology makes it possible to implement such control using oxygen sensors based on solid oxide electrolyte [7].

**Practice:**

Three important issues should be considered for the design of the mass exchanger.
1. The stability of the solid–phase oxygen source.
2. The efficiency of electric heaters working under the condition of temperature variation from 400-650 °C and duration of 1000-5000 hours.
3. The consideration of the pump characteristics provided (increase of coolant flow rate) inside the mass exchanger.

Use of pressed lead oxide spheroids as solid-phase source or dissolved oxygen has been justified and will be obtained with the collaboration with Los Alamos National Laboratory and John Farley’s team in UNLV. Testing of this material under static conditions showed no mechanical damage of PbO spheroids, which were preliminarily annealed in air at 600 degree C and coated with lead. Neither spallings, nor racks were revealed on the surface of spheroids and no fragments of spheroids were revealed on the surface of liquid lead. Besides that the thermal stability of solid-phase source of dissolved oxygen was confirmed experimentally.

Particularly, in the real circuit, there is a possibility of oxidizer “poisoning”, formation on its surface of compounds, based on structural material components (Fe and to a lesser extent Cr). This oxidizer “poisoning” will block the oxygen dissolving process. This effect is caused by a chemical interaction of iron impurity with the surface layer of lead oxide. Layer thickness is 0.05mm to 1mm depending on the time and the conditions of the interaction. The oxidizer “poisoning” causes 4~5 times decrease of the rate of their dissolution in the liquid metal at the temperature of about 620 °C. This drawback can be prevented by returning some part of oxidized coolant (10-15% of its volume) leaving mass exchanger to strongly deoxidized liquid metal flow entering back to the mass exchanger.

Planned work/tasks:

I. Oxygen sensor and flow rate sensor fabrication

The oxygen thermodynamic activity automatic controlling system uses the oxygen activity sensors located in several areas of the circuit (including “cold” part) Fig 5. The comprehensive approach to the control action should include an analysis of the readings of the oxygen sensors along with the temperature and flow rate sensors.

The oxygen sensor, however, needs to be calibrated in advance for oxygen concentration dissolved in liquid lead/alloy. Based on the research work of task-13 (TRP program), the following problems regarding to the oxygen sensor should be solved additionally in the process of each development:

a) One set of oxygen sensor testing and calibration facilities should be set up at UNLV,
b) Fabrication of sensor as a whole,
c) Testing and calibration of this oxygen sensor.

As a result of it, the calibrated sensors will be used for tests to develop an automatic control system of oxygen thermodynamics activities.
II. Mass exchanger design

The simulation regarding the heat and mass transfer of the liquid lead/alloy will be carried out for better understanding of the mass exchanger. The optimized porous medium for passing and interacting with the liquid lead/alloy will be studied based on the simulation of the flow characters in porous medium with surface reaction and the exchange rate. The mass exchange rate depends on temperature in reaction vessel and coolant velocity in the mass exchanger vessel.

The mass exchanger designs will be tested in specially prepared facilities (circuit and static facilities) first in the manual control mode (by the operator), and then using the oxygen TDA automatic control system under condition of about 1000 hour continuous operation, at a temperature range of 400 °C to 550 °C with a specified oxygen TDA range of E=350-370 mv (Oxygen sensor signal reading).

![Oxygen controlling system](image)

**Fig. 5** Oxygen controlling system
III. Solid-phase oxygen automatic controlling system

A technical implementation of solid-phase control method is possible using specially designed devices, mass exchangers, which are the most important components of the complex of the heavy metal coolant technology (see Fig. 4).

The automatic control algorithms (for example, PID scheme) will be developed based on the signal response of sensors, flow meters, thermocouples to maintain oxygen concentration in the level of corrosion protection. In addition, it is necessary to determine algorithms for verification of readings of oxygen sensors and algorithms for the control of their integrity and efficiency, i.e., the simulation for control algorithms will be carried out before it is applied to the real oxygen control system.

IV. Facilities used for the loop-type test and static test

The modification of the present existing lead-bismuth loop will be developed for the testing of solid-phase oxygen controlling system

V. Optimization of Porosity

A new design scheme of mechanical stable solid phase mass exchange device with optimized porosity for fully interaction between the solid and the passing liquid is proposed. We will first develop a numerical fluid dynamics code of flow in porous medium with chemical reaction. The mass exchange rate, the interaction between the solid and liquid phases and the solid phase mechanical characters will be determined based on the code. Then an initial solid lead oxide exchange device will be designed and experiments will be carried out. Optimization study will be applied to the model to improve its performance. Additionally, through comparing simulations and experiments, the device will be optimized. Finally, we will design a solid phase oxygen control system that can be applied to practical lead-alloys coolant system.

4. Reference:

5. Research Objectives and Goals:

The research objectives are:

Justification of solid-phase method of oxygen TDA control in lead-containing coolants and mass exchanger designs for its implementation under static and dynamic conditions.

The research goals are:

- Address key open questions about the oxygen control system applied to control of the oxygen concentration in practical liquid lead and lead-bismuth eutectic nuclear coolant systems.
- Design a mass exchanger with solid-phase oxygen automatic control system that can bridge the present gaps between tests and practical applications.

6. Technical Impact:

The impact of a successful project will be a breakthrough, which will achieve a control system of the oxygen level using a solid-phase control system without any slag deposition. The results of proposed project will make it possible to use “oxygen” technology for assuring efficient and reliable operation of research and commercial facilities with lead coolant. The model and method could become of critical value for many DOE programs.

7. Research Approach:

A 3-D finite element (FE) code of flow in porous medium with chemical reaction will be developed. The mass exchange rate, the interaction between the solid and liquid phases and the solid-phase mechanical characters will be determined based on the code. Then an initial solid lead oxide exchanger will be designed and verification experiments will be carried out. An optimization study as well as the control simulation and scheme will be applied to the oxygen automatic controlling system to improve its performance. Finally, one solid-phase-oxygen controlling mass-exchanger will be designed and can be applied to practical lead-alloys coolant system.
8. Expected Technical Results:

- A detailed theoretical and experimental understanding of the thermodynamics, flow characteristics, solid mechanics stability, and interactions of the solid porous mediums and the passing liquid lead/alloy coolant with low Prandtl number in porous medium with surface reactions;
- Numerical modeling of thermodynamics, flow characteristics and mass transport of liquid lead/alloy with solid-phase PbO spheroids;
- Technology of manufacturing of solid-phase PbO spheroids as sources of oxygen in mass exchangers, their components and mass exchanger design as a whole;
- Manufacture of mass exchanger with built-in heaters, pump and their combination;
- Creation of systems of oxygen controlling for static and dynamic test facilities with lead coolants;
- Publications and reports on the results.

9. Capabilities at the University and Los Alamos:

The oxygen sensors, gas control system, temperature controller, as well as the sensor testing facilities are available in UNLV, which is under TRP project of Task 13 (Yingtao Jiang’s team)

The experimental space with hood will be ready in Chemistry department with the collaboration of John Farley’s team. Besides, one gas phase oxygen controlling system is available.

Other facility would be available in Los Alamos National Laboratory (LANL) with the collaboration of Dr. Ning Li’s Advanced Fuel Cycle Initiative (AFCI) program.

10. Project Timeline:

Timeline Narrative

The proposed research is planned to cover three years, starting in Fall 2005 (at the beginning of the academic year).

Milestones

- Completion of numerical modeling of thermodynamics, flow characteristics and mass transport of liquid lead/alloy with solid-phase PbO spheroids; (Summer, 2007)
- Completion of mass exchanger design; (Summer, 2007)
- Completion of automatic controlling system design; (Winter, 2007)
- Completion of justification of solid-phase oxygen automatic control system. (Summer, 2008)

Deliverables:
• Collaboration with DOE project: Monthly communications (by phone or in person) with LANL project collaborator and/or technical lead to update on progress, discuss problems.
• Progress Reports: Brief reports indicating progress will be provided every quarter.
• Bi-annual Reports: Written reports detailing experiment and simulation performed, and data collected, and results to date.
• Final Report: Written report detailing experiments and simulation performed, data collected, results, and conclusions to be submitted at the end of the project.
• This project will lead to one Ph.D. dissertation and at least two M.S. theses from the graduate students participating in this project.

11. Work Proposed for Academic Year 2005-2006, Goals and Expected Results:

A detailed study will be carried out regarding to the theoretical and experimental understanding of the thermodynamics, flow characteristics, and the interactions of the solid porous mediums and the passing liquid lead or LBE. The numerical code for simulation will be developed. The mass exchanger design will be carried out as well as supporting experiments for validation of the solid-phase oxygen automatic control system.
12. Biographical Information:

**Jian Ma:** will be a research assistant Professor after April, 2005 of Mechanical Engineering at the University of Nevada, Las Vegas. Now, he is a research scholar of Mechanical Engineering at UNLV. He received his B.S. and M.S. degree in Fluid Mechanics at Fudan University, Shanghai, P. R. China and received his Ph.D degree in Nanyang Technological University, Singapore. His research interests include experimental study on thermal and fluids research, non-intrusive fluid dynamic diagnosis, Micro-fluids, Bio-chips as well as the control devices and instrumentation. He carried out the theoretical and experimental research on the extremely-low-level oxygen sensor development in the application of liquid lead and lead bismuth eutectic (LBE) in UNLV and Los Alamos National Laboratory. He accomplished the simulation of enhancement of oxygen transfer in LBE using natural convection. Now he is the principle investigator in the project of LBE TC-1 loop in UNLV.

**Mohamed B. Trabia:** is a Professor and the chairman of Mechanical Engineering Department at the University of Nevada, Las Vegas. He received his B.S. and M.S. in Mechanical Engineering at Alexandria University, Alexandria, Egypt and completed his Ph.D. degree in Mechanical Engineering of Arizona State University. His research interests include
- Kinematics Analysis and Design of Robots
- Dynamic Analysis and Control of Flexible Robots.
- Path Planning and Obstacle Avoidance of Mobile Robots.
- Vibration Analysis of Fans.
- Optimization Applications to Engineering Design.
- Fuzzy Logic Control Applications.

**Samir Moujaes:** is a Professor of Mechanical Engineering at the University of Nevada, Las Vegas and will serve as Principal Investigator for this project. Prof. Moujaes has worked for five years on computational aspects of cooling of canisters for the Yucca Mountain Project for DOE using FIDAP CFD package. Two emplacement configurations of high level waste containers were investigated and the temperature profiles and air velocity profiles under natural convection conditions were calculated. Other computational work involved developing two- and three-dimensional models for the description of heat transfer processes in residential gabled attics under the influence of the three modes of heat transfer. Currently a model is also being developed to describe the interaction of this heat transfer on the heat pickup of the supply air through a typical attic placed supply duct. Other impending projects have to do with the newly established National center for Energy Management and Building Technologies (NCEMBT) established at UNLV whose funding is going to be around $2.4M for 03-04 of which Dr. Moujaes will be involved in about $600K expenditure of that funding. A new project that is coming on line is the investigation of the potential of high temperature thermo-chemical production of hydrogen by solar means. UNLV is involved in an overall effort for 03-4 of over $2.0M out of which UNLV will obtain about $670K and out of which Dr. Moujaes will obtain a funding of approximately $75K. Another high temperature hydrogen production research project involving an advanced nuclear reactor technology is in the process of being evaluated and where the PI is involved in the total funding there is about $2.0M for three years. The PI will be funded
for about $100K for the first year. Other experimental work has involved two-phase flow hydrodynamics and the determination of profiles of localized values of void fraction and dispersed phase axial velocity through the use of locally developed dual-tipped fiber optics probes. Prof. Moujaes has also been involved with R&D on the testing for three-phase hydrodynamics and heat transfer of slurry derived from coal for a Solvent Refined Coal (SRC-I) process. He has published several papers in ASME, ASHRAE, and the Journal of Energy Engineering and is a reviewer for these organizations. He is an Associate Editor for the JEE and has also organized and chaired sessions in some of their conferences.

**Yingtao Jiang:** is an Assistant Professor of the Department of Electrical and Computer Engineering at the University of Nevada, Las Vegas. He received his B. Eng. degree in Biomedical Engineering and Electronics at the Chongqing University, Chongqing, China in 1993, and M. A. Sc. degree in Electrical Engineering at Concordia University, Montreal, Canada in 1997. Dr. Jiang completed his Ph. D study at the University of Texas at Dallas in 2001. His research interests include algorithms, VLSI architectures, and circuit level techniques for the design of DSP, networking, and telecommunications systems, computer architectures, and designs of sensor-based data acquisition systems. He has been serving as the PI in TRP Task 13.

**Jinsuo Zhang:** is Research Assistant Professor of the Department of Mechanical Engineering at the University of Nevada Las Vegas and Research Associate in the Condensed Matter and Thermal Physics Group at the Los Alamos National Laboratory, and would serve as Co-Principal Investigator. Dr. Zhang obtained his Ph.D. degree from Zhejiang University in December 2001, His research in the fields of fluid mechanics and corrosion science for the past 6 years is truly outstanding and has resulted in more than two dozen papers in top peer review journals such as International Journal of Heat and Fluid Flow, Journal of Hydrodynamics, Physical Review E, Journal of Nuclear Materials, and Nuclear Technology. Dr. Zhang is an expert in the corrosion model field of lead-alloys technology. He has published several journal papers on the lead-alloy technology. He is now one of the key members of the lead-bismuth development technology team in the Los Alamos National Laboratory.
From: Ning Li, Ph.D.  
Project Leader, Lead-Alloy Coolant Technology Development, AFCI  
Subject: Support Statement

To Whom It May Concern:

This letter is provided in support of the proposed project “Solid-Phase Oxygen Control System”, for which I’ll serve as a national lab collaborator.

In lead-alloy coolant technology, it is critically important to measure and control the oxygen concentration in the liquid metal to mitigate steel corrosion and coolant contamination. The necessity and efficacy of oxygen control have been demonstrated in worldwide R&D. However, the existing implementations, mostly based on gas-phase control, have some significant drawbacks both in the low transfer and adjustment rates, and in localized slag formation at the gas/liquid metal interface. LANL has started an experimental implementation of a PbO-based solid mass exchanger for DELTA to improve the control performance.

This proposal will investigate the mechanisms, kinetics and control issues of the solid-phase oxygen control systems. The success of this project will help establish the scientific and technological basis of this new approach. The discoveries and improvement resulting from the research will enhance the performance of such implementation in DELTA, directly supporting an important experimental facility in the national program where resource and funding limitation does not allow this R&D. The project is also synergistic to other related TRP tasks, and in particular the TC-1 LBE target loop operation and future upgrade and transformation. I believe that this proposal meets the UNLV TRP objectives. I strongly support this project and will actively engage in the collaboration.

Yours sincerely,

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(Ning Li)