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Theoretical Modeling of Protective Oxide Layer Growth in Non-isothermal Lead-Alloys Coolant Systems

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Project Title: Theoretical Modeling of Protective Oxide Layer Growth in Non-isothermal Lead-Alloys Coolant Systems

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TRP Research Area: Transmutation Sciences Technology/Coolant Technology

Proposed Budget: $ 151,941

Project Period: September 1, 2004 – August 31, 2005

Submitted Date: April 30, 2004

Abstract

The goal of the proposed research project is to provide basic understanding of the protective oxide layer behaviors and to develop oxide layer growth models of steels in non-isothermal lead-alloys (lead or lead-bismuth eutectic) coolant systems. It is widely recognized that the corrosiveness of the lead-alloys is a critical obstacle and challenge for which it can be safely used or applied in the nuclear coolant systems. Active oxygen control technique can promote the formation of the "self-healing" oxide films on the structural material surface, drastically reducing steel corrosion and coolant contamination. Many experiments of steels exposed to flowing lead-alloys have been carried out to study the protective oxide layer behaviors. However, the experimental data are still very incomplete at present and can not provide the dependence of the oxide behaviors on the system operating temperature, temperature profiles along the lead-alloys loop, oxygen concentration, flow velocity, etc. In addition, oxygen distribution in a non-isothermal lead-alloys coolant system is not well understood. Precise studies and simulations of all hydrodynamics with thermal conditions encountered in practical coolant loop systems by use of different flowing conditions in the laboratory are difficult and expensive, if not impossible. Therefore it is important and necessary to develop theoretical models to predict the protective oxide layer behaviors at the design stage of a practical lead-alloys coolant system, to properly interpret and apply experimental results from test loops, and to provide guidance for optimization in lead-alloys nuclear coolant systems. The research project, therefore, is aimed at filling the gaps of protective oxide layer growth and the oxygen concentration level before lead-alloys nuclear coolant is ready for programmatic implementations and industrial applications. In addition, the project will lead to one Ph.D. dissertation and one M.S. thesis from the graduate students participating in this project and at least three journal papers.

Work Proposed for Academic Year 2004-2005, Goals, and Expected Results

Surface reaction kinetics, the transport process of oxygen, and the corrosion products though the multi-phase layer will be first studied and modeled. Difference between the oxidation of steel in lead-alloys eutectic and that in pure air will be identified. An oxide growth model of steel in static lead-alloys will also be developed. The model will be applied to study the dependence on temperature, oxygen level and steel chemical compositions. We also plan to study the oxygen distribution in non-isothermal system based on a kinetic corrosion model [1]. The students will attend relevant technique conferences to present their research results.

Funding Profile

Academic Year:	**2004-2005**	**2005-2006**	**2006-2007**
Total:	**$151,941**	**$151,941**	**$151,941**

Background and Rationale

In ATW (Accelerator-driven Transmutation of Waste) and Generation IV Nuclear Energy Systems technology evaluations, lead-alloys (lead, lead-bismuth eutectic) emerge as strong candidates for transmutation and advanced reactor systems as nuclear coolants and high-power spallation neutron targets. However, it is widely recognized that corrosion of materials caused by lead-alloys presents a critical barrier to their industrial use. A few experimental research and development projects have been set up by different groups such as LANL to study the corrosion phenomena in their test facilities and to develop mitigation techniques and materials [2].

One of the central or main techniques in lead-alloys coolant technology that has been under developing is to use the active control of oxygen thermodynamic activity (OTA) to provide protective oxide layers [3]. Setting OTA in flowing lead-alloys makes corrosion highly dependent upon the oxygen concentration and the oxidation processes at materials surfaces. The active oxygen control technique exploits the fact that lead and bismuth are chemically less active than the major components of steels, such as *Fe, Ni,* and *Cr*. By carefully controlling the oxygen concentration in LBE, it is possible to maintain an iron and chrome based oxide film on the surfaces of structural steels, while keeping lead and bismuth from excessive oxidization that can lead to precipitation contamination [4]. Thermal analysis has given an idea oxygen level range in a non-isothermal lead-alloys coolant system [3]. However, in a practical coolant loop, the proper oxygen level depends not only on thermal factors but also on hydraulic factors (system operating temperature, temperature profile, flow velocity, etc.). In addition, the oxygen distribution in a non-isothermal lead-alloys coolant system is still unclear. The optimal oxygen levels still need to be investigated.

Several experimental studies addressing on the corrosion and oxidation issues in oxygen control lead or lead-bismuth systems have been published in the near past three years [5-8]. These works indicate that the temperature, oxygen concentration, flow velocity, materials composition, etc. play important roles on the oxidation processes of steel in LBE. The kinetic corrosion models [1,4, 9,10] indicate that the oxidation process depends on both local and global conditions of the entire system. However, the previous experimental studies are almost focus on the oxide layer behaviors at the test leg. The test data can not be directly applied to the other legs in the same loop and to a loop with different global conditions. At the present time, the expensive and scarce test results can not be easily interpreted and applied for general design purposes. We can not reach a general conclusion or a correlation between the protective oxide layer growth and the hydraulic factors and oxygen level based on the present experimental results.

There are many models on the oxide growth for steels in air [11]. The main difference between the two systems (steel in air and steel in lead-alloys with oxygen control) is that metals, such as iron (main component of steel), can enter into the liquid metal through diffusion of the metal and reaction between the lead and the oxide even after the oxide is formed. On the words, dissolution and oxidation of steel components occur simultaneously in lead-alloys systems. Therefore, the oxide layer behaviors of steels in lead-bismuth eutectic (LBE) are more complex than that in air. To understand the oxide layer growth behaviors in a non-isothermal flow lead-alloys coolant system, the dissolution, the hydraulic factors' effects must be included.

Precise simulations of all hydrodynamic and thermal conditions encountered in practical coolant loop systems by use of different flowing conditions in the laboratory are difficult and expensive,

if not impossible. Therefore it is important and necessary to develop theoretical models to predict the protective oxide layer behaviors at the design stage of a practical lead-alloys coolant system, to properly interpret and apply experimental results from test loops, and to provide guidance for optimization in lead-alloys nuclear coolant systems. This research project, therefore, is aimed at filling the gaps of protective oxide layer growth and the oxygen concentration level before lead-alloys nuclear coolant is ready for programmatic implementations and industrial applications. Two graduate students will be trained in this field and the project will lead to one Ph.D. dissertation and one M.S. thesis.

Research Objectives and Goals

The research objectives are:

- To elucidate the mechanism of the protective oxide layer growth of steels in static, non-isothermal flowing lead-alloys coolant systems with oxygen concentration level control.
- To elucidate the mechanism of mass transport of oxygen, corrosion products in the multi-phase system.
- To develop oxidation growth models of steels in lead-alloys coolant systems.
- To clarify the dependence of oxidation process on the hydraulics factors (system operating temperature, temperature profile, flow velocity, etc) and the oxygen concentration distribution and level.
- To clarify the optimal oxygen concentration levels in practical coolant system scales.
- To interpret the experimental results from test loops and to apply them to the design of practical nuclear coolant systems.

The research goals are:

- To understand the difference in oxidation behaviors between different types of structure materials.
- To incorporate the present oxide layer growth model to our previous kinetic corrosion model.
- To develop a general numerical code that can predict the oxygen concentration level, the oxidation growth rate and the corrosion rate in practical lead-alloys coolant systems.
- To advance the overall understanding of corrosion/oxidation in lead-alloys systems.

Technical Impact

The proposed work has great importance and will make a major contribution to the lead-alloys (lead, lead bismuth eutectic) technology that is being developed for high-power spallation neutron targets and nuclear coolants. Active oxygen control technique is one of the techniques to mitigate corrosion in lead-alloys systems. Experimental results are too incomplete to apply in the coolant system design. The theoretical oxide growth models obtained from this research project can interpret the scare experimental data and apply them to design of practical lead-alloys coolant systems. After successfully completing the project, we will develop and understand fundamental theories on the oxide growth mechanisms and the dependence on the thermal and hydraulic factors. The optimal oxygen concentration level in a non-isothermal nuclear coolant

loop scale will be identified. An analysis tool for the calculation of the oxygen concentration level and the oxygen consumption will also be developed. In addition, the proposed research provides important insight to the design, operation and testing of lead-alloys loop systems.

Research Approach

(1) Preliminary studies on the oxygen transport in non-isothermal lead-alloys loop reveals different solution regimes and important control parameters:

Theoretical analysis of the oxygen transfer process is possible for several simple cases. For example, by assuming homogeneity of oxygen in the bulk fluid due to the turbulent flow and adopting the approximation of the diffusion layer at the oxide layer interface, we have conducted a few preliminary studies on kinetic modeling of corrosion processes and have derived a simplified kinetic model [1]. This model has successfully simulated laboratory experiments and final solution classification, key parameter identification, and sensitivity studies. However, more details of research and development are needed and performed to extend their usage for oxygen transport in "full-scale" LBE systems.

(2) Theoretical study on the protective oxide film growth in static liquid lead-alloys reveals the oxidation characteristics of steels in liquid lead-alloys with oxygen concentration level control:

According to some experimental results [6], the protective oxide layer on steel structures in an oxygen controled LBE system is composed of an external layer (Fe_3O_4) and the internal spinal layer. There are many references [11] on the modeling of the two layers for steels exposed to air. The main difference between the two systems (steels in air and steels in lead-alloys with oxygen concentration level control) is that the steel components can dissolve into the liquid lead-alloys. We will extend the existing oxide formation model first developed by Wagner [11] to a model that includes both dissolution and oxidation at surfaces of steels in stationary liquid lead-alloys.

(3) Development of oxide layer growth model in flowing lead-alloys reveals the flow velocity effects:

In a flowing system, the flowing lead or lead-bismuth eutectic (LBE) can quickly transport the oxygen to steel surfaces, which results in a high oxidation rate. On the other hand, the corrosion product can be taken away quickly from the surface by convection, which reduces the oxide layer thickness. The protective oxide layer behaviors in flowing system should be significantly different from that in the static system. Extending the oxide growth model in the static lead-alloys system to the flowing system will reveal the effects of flow velocity on the oxidation growth rate.

(4) Modeling of oxygen and corrosion products transport process reveals the oxygen distribution in non-isothermal lead-alloys coolant loop systems:

Once the protective oxide layer is formed, the transport of oxygen and corrosion products such as iron can be depicted as Figure 1. In the solid (bulk steel and oxide layer), oxygen profile is determined by both diffusion and surface reaction; in the mass transport boundary layer, the

oxygen profile is determined by both convection and diffusion, while in bulk lead-bismuth eutectic (LBE) or lead, the convection dominates the transport process. Theoretical analysis will obtain a theoretical solution of the mass transport equations, which provide information on the oxygen distribution in the entire coolant loop.

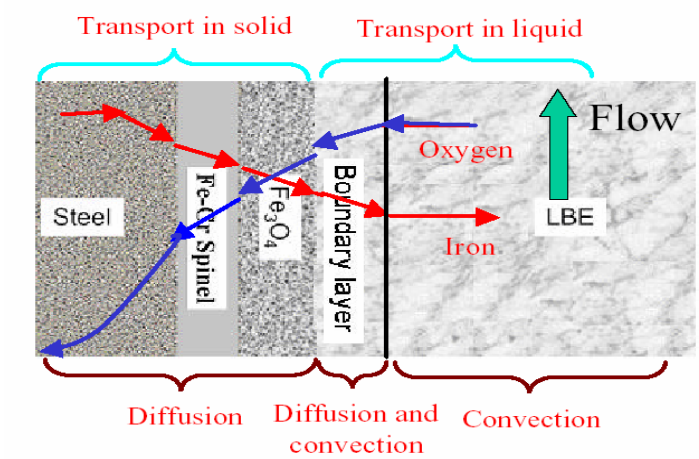

Figure 1. Depiction of the oxygen controlled corrosion product transport process

(5) Correlations between the oxide layer growth rate and hydrodynamic conditions:

In non-isothermal lead-alloys coolant systems, the protective oxide layer thickness depends on both local and global conditions of the entire loop. The oxide layer growth model in a coolant loop scale will be used to predict the local oxide growth rate regarding the global conditions. Based on comparisons of theoretical results and the experimental results from DELTA loop (a material test loop designed and established at LANL). The experimental results will be interpreted. Correlations between the oxide growth rate and the hydrodynamic factors (velocity, local temperature, temperature profile, oxygen concentration level, etc) will also be developed to predict the protective oxide layer behaviors at design stage of the practical lead-alloys coolant system.

(6) Analysis tool for the calculation of the oxygen concentration level and the oxygen consumption:

In a non-isothermal flowing lead-bismuth eutectic (LBE) system, the oxygen concentration should be kept at a certain level so that the oxide layer can be formed in the hottest temperature leg while no lead oxide precipitates in the coldest leg. The controlling phenomenon is the oxygen transport kinetics that depends on the diffusion, convection, and the chemical reaction. Based on oxide growth model and the oxygen transport model, the optimal oxygen levels are identified. A general analysis tool will be developed to calculate the oxygen level, distribution, consumption, the oxide layer growth rate and the oxide layer thickness after the system reaches the steady state.

(7) Interpretation of experimental results from Delta loop at Los Alamos National Laboratory will provide guidance for optimization in lead-alloy nuclear coolant system:

Comparisons between the modeling results and the experimental results from Delta loop at the Los Alamos National Laboratory will not only verify the theoretical model but also provide important information on the oxide growth mechanism in flowing lead-alloy system. The oxide growth rate and the scale removal rate by the flowing lead-alloy will be obtained by fitting the experimental results using the theoretical model, which will provide guidance for optimization in lead-alloy nuclear coolant system. Table 1 shows the preliminary comparisons of the preliminary model results and some experimental results of US steels from TC-1 LBE loop at IPPE.

Steel	$P_{O_2}^{I}$ (atm)	c_O (ppm)	$P_{O_2}^{II}$ (atm)	K_p (m^2/s)	Time(h)	Oxide Thickness(μm)	Experimental results		Error
316 at 550°C	1.70 $\times 10^{-27}$	0.03	1.19 $\times 10^{-21}$	2.29 $\times 10^{-17}$	1000	17.25	Time (h)	Oxide thickness(μm)	
					2000	24.40	1000	4-10	142%
					3000	29.88	2000	16-20	33%
		0.05	3.32 $\times 10^{-21}$	2.30 $\times 10^{-17}$	1000	17.30	3000	10-32	42%
					2000	24.47			
					3000	29.97			
D-9 at 550°C	1.63 $\times 10^{-27}$	0.03	1.19 $\times 10^{-21}$	2.36 $\times 10^{-17}$	1000	17.50	1000	5-20	40%
					2000	24.74	2000	20-36	11.6%
					3000	30.30	3000	12-40	16.5%
		0.05	3.32 $\times 10^{-21}$	2.37 $\times 10^{-17}$	1000	17.55			
					2000	24.81			
					3000	39.39			
HT-9 at 550°C	1.24 $\times 10^{-27}$	0.03	1.19 $\times 10^{-21}$	2.83 $\times 10^{-17}$	1000	19.18	1000	20	4.1%
					2000	27.12	2000	32-36	20%
					3000	33.21	3000	12-38	32.8%
		0.05	3.32 $\times 10^{-21}$	2.84 $\times 10^{-17}$	1000	19.22			
					2000	27.18			
					3000	33.29			
HT-9 at 460°C	5.94 $\times 10^{-32}$	0.03	9.64 $\times 10^{-24}$	4.15 $\times 10^{-19}$	1000	2.32	1000	1-8	48%
					2000	3.28	2000	12-14	75%
					3000	4.02	3000	14-16	73%
		0.05	2.68 $\times 10^{-23}$	4.80 $\times 10^{-19}$	1000	2.50			
					2000	3.53			
					3000	4.32			

Table.1 Preliminary comparison between the calculation results and experimental results [12]

<u>(8) Pre-oxide analysis provides powerful roadmap to study material performance in lead-alloy technologically important applications.</u>

Pre-oxidation is one effective method to set the protective oxide layer. The initial condition will be classified into distinct categories. For the present proposed studies, the combined effects of the oxidation and the scale removal by the flowing lead-alloy (mass transfer corrosion or erosion) will lead to a limited oxide thickness. The thickness has to be structurally stable for the entire process to proceed. Pre-oxide analysis will show how the oxide thickness and weight change data are used, what simplified model is applicable in what range with what initial conditions. Fig.2 shows the expecting oxide thickness variations [13].

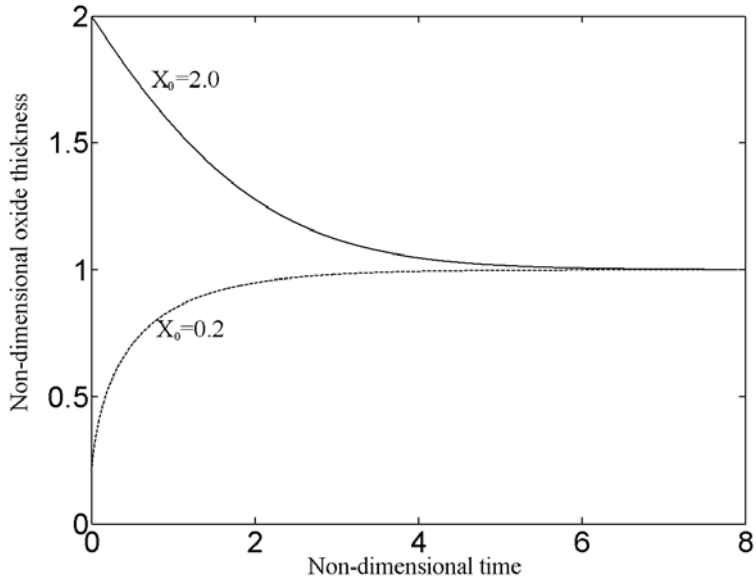

Fig.2 Expecting oxide thickness variation with time in flowing oxygen controlled flowing lead-alloy systems. The thickness is normalized by the limited oxide thickness.

Expected Technical Results

The following scientific and technical results will be delivered after three-year proposed research:

1. Illustration of the oxidation process mechanisms in oxygen control lead-alloys systems
2. Development of an efficient theoretical kinetic model for oxidation process in non-isothermal lead-alloys coolant system.
3. Identification of the protective oxide layer growth rate and the dependence on the thermal and hydrodynamic factors of the entire coolant loop.
4. Optimal operation conditions for oxygen control lead-alloys systems.
5. Optimal pre-oxide conditions
6. Analytical models for various limiting process regimes.
7. Development correlations and tools for calculations of the oxidation rate, oxygen concentration level and distribution, and oxygen consumption.
8. Publications in peer-reviewed journals

Capabilities at the University of Nevada, Las Vegas and the Los Alamos National Laboratory:

Dr. Yitung Chen is Associate Professor of the Department of Mechanical Engineering and Associate Director of the Nevada Center for Advanced Computational Methods (NCACM) at the University of Nevada Las Vegas, and would serve as Principal Investigator. He received his B.S. degree in Chemical Engineering in 1983, and his M.S. and Ph.D. degrees in Mechanical Engineering in 1988 and 1991, respectively, from the University of Utah. He also has a minor degree in Nuclear Engineering. He was a consultant for several engineering companies from 1991 to 1993. Dr. Chen is an expert in experimental and computational aspects of momentum,

heat, and mass transfer. His research interests include chemical kinetics modeling, high level radioactive waste repository design, atmospheric sciences, magnetohydrodynamics modeling, ground water transport, energy conservation, and biomedical engineering. He also has a strong background in organic chemistry, biochemistry, polymer chemistry, and physical chemistry. His research experience includes being PI and co-PI on projects involving the study of flow and heat transfer and species transport in unsaturated porous media funded by DOE, the Transmutation Research Program-University Participation Program funded by DOE, the high temperature heat exchanger design funded by DOE, the Solar Thermal Chemical Hydrogen (STCH) Generation funded by DOE, the burning of rocket motors under the Joint Demilitarization Technology (JDT) program funded by DOD, Radiography Stockpile Stewardship Program funded by DOE, ATLAS project funded by DOE, JASPER project funded by DOE, high-level radioactive waste material repository design funded by DOE, high performance computing project funded by NSF, and atmospheric modeling funded by the NOAA Cooperative Institute for Atmospheric Sciences and Terrestrial Applications. He is also co-PI on an EPA project dealing with environmental monitoring for public access funded by EPA and a groundwater modeling project funded by DOE.

Dr. Jinsuo Zhang is Research Assistant Professor of the Department of Mechanical Engineering at the University of Nevada Las Vegas and Research Associate 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.

Dr. Jichun Li is Assistant Professor of the Department of Mathematics at the University of Nevada Las Vegas. He is a Computational Mathematician; his research has been focused on numerical analysis, mathematical modeling and parallel computing, as evidenced by his over 25 refereed journal publications. Dr. Li would serve as Co-Principal Investigator. He has extensive experience in scientific computation using Fortran, C/C++, and MPI. He has been involved with multidisciplinary research ever since he worked at the Center for Subsurface Modeling (lead by Professor Mary Wheeler) at the University of Texas at Austin during May 1998 to August 2000. His completed package UTPROJ (in C++ and MPI) is still used by U.S. Army Engineer Research and Development Center at Vicksburg, Mississippi. Since 2000, Dr. Li constantly teaches courses such as numerical methods and high-performance computing at UNLV. Apart from his teaching activities, Dr. Li actively continues his research work on different topics involving finite element method, meshless method, environmental modeling on both surface water and groundwater, image processing, and parallel computing.

Dr. Ning Li is the team leader and technical staff member in the Advanced Fuel Cycle Initiative (AFCI) Program at the Los Alamos National Laboratory, New Mexico, and an Adjunct Professor of Mechanical Engineering and the International Programs Coordinator for the Transmutation

Research Program (TRP) at the University of Nevada, Las Vegas. He received his Ph.D. in Physics from the University of California, Santa Barbara. He has over 14 years of research and program development experience in nonlinear dynamics, separation technologies, and liquid metal nuclear coolant and high power spallation target technology for advanced reactors and transmutation systems. Dr. Li has published over 60 technical papers and reports. Dr. Li served as project leader, principal investigator and technical leader for over $12M in Laboratory and federal research funds. He is a member of the American Nuclear Society, an executive member of the Materials Science and Technology Division, and serves on various Laboratory and US Depart of Energy (DOE) committees for proposal reviews and program oversight. Dr. Li participated and led sub-task groups to develop two DOE national R&D roadmaps for Accelerator-driven Transmutation of Waste (ATW) and Generation IV Nuclear Energy Systems (Gen IV), which were presented as reports to Congress and led to the formation of the national programs.

Extensive computing facilities exist for the modeling efforts at UNLV. Computing facilities range from workstations to super computers. These facilities will be available for the analysis and design of the proposed concepts. A wide range of computational tools exists on these systems.

Project Timeline for the first year (2004-2005)

This proposal describes a research program that will take three years to be completed. The timeline describes the expected milestones, and deliverables for the first year only (2004-2005). At the end of the first year, separate proposals will be submitted for the second and third year.

Milestones (Based on starting date of May 15th, 2004)

- Analysis of the oxygen distribution at a steady state in non-isothermal lead-alloys coolant loops. Development of a diffusion control model to analysis the dependence of oxygen distribution on the global temperature profile (August, 2004)
- Analysis the oxygen behaviors in the DELTA loop using the developed model (September, 2004)
- Development of an oxide layer growth model for pure iron, steel in static liquid lead-alloys (December, 2004)
- Analysis of the dependence of oxide layer rate on the oxygen level, temperature and development of correlations in static conditions (March, 2005)
- Carry out initial study of the oxide behaviors in dynamic system (May, 2005)
- Preparation of annual report (May, 2005)

Deliverables

In addition to the monthly updates and quarterly and final reports, we expect to publish the results of this project in peer-reviewed journal and the appropriate technical conferences (the students will attend). The project will lead to one Ph.D. dissertation and one M.S. thesis from the graduate students participating in this project and at least three journal papers.

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