Form B: IGEM-HERC Full Proposal Cover Sheet

Idaho State Board of Education

PROPOSAL NUMBER: TOTAL AMOUNT REQUESTED: \$197,600

Proposal Track (select one): Proof of Concept

TITLE OF PROPOSED PROJECT: Self-healing Composites for Aggressive Environments

SPECIFIC PROJECT FOCUS: The proposed research focuses on developing advanced materials that can self-heal defects for applications in next-generation power plant components, including nuclear reactors, specifically modular and microreactors. The self-healing materials can also be used in the currently operating LWR nuclear reactors and other power plant components.

PROJECT START DATE: July 1, 2024	PROJECT END DATE: December 31, 2025				
NAME OF INSTITUTION: University of Idaho	DEPARTMENT:	Nuclear	Engineering	and	
	Industrial Management				

ADDRESS: 1776 Science Center Drive, Idaho Falls, ID 83402

E-MAIL ADDRESS: ksraja@uidaho.edu		PHONE NUMBER: (208) 757-5406			
NAME:	TITLE:		SIGNATURE	:	
PROJECT DIRECTOR/PRINCIPAL INVESTIGATOR	Krishnan Raja	Professor	- Krich 5 #	linge	
CO-PRINCIPAL INVESTIGATOR	Indrajit Charit	Professor	Indraji	7 Charit	
NAME OF PARTNERING	COMPANY REPRESENTATIVE NAME:				
SIGNATURE:					
Authorized Organizational	NAME: Heather Clark Director, Sponsored Acc	, Asst. SIGN ounting,	IATURE:		
Representative	University of Idaho				

ee 3/12/24

Proposal Narrative

Idaho Public Institution	University of Idaho (UI)				
Project Title	Self-healing Composites for Aggressive Environments				
Principal Investigator	Krishnan S Raja, Professor, Nuclear Engineering and Industrial Management, University of Idaho, Idaho Falls, ID-83402 (ksraja@uidaho.edu)				
<u>Co-PI</u>	Indrajit Charit, Professor and Chair, Nuclear Engineering a Industrial Management, University of Idaho, Idaho Falls, ID-834 (<u>icharit@uidaho.edu</u>)				

Project Objectives

Self-healing bulk composite materials will be developed using a powder metallurgy route and sintering process, showing self-healing of micro-cracks, superior resistance to crack initiation, irradiation damage, and corrosion. Site-specific precipitation of solute atoms will be the mechanism for self-healing of irradiation and creep damages. Microscopic damage to coatings will be healed by distributing core-shell configured nano-capsules of metal-core/oxide-shell (MCOS). Healing of a propagating crack occurs when the micro-capsules are sheared, and the metal core fills the crack. The novel materials and manufacturing steps developed in this project will help improve the reliability and safety of the components used in current and next-generation nuclear reactors and other high-temperature applications. In this proposed research, we will employ a two-pronged approach. In approach one, Fe-B-C nano-dispersoids will be mechanically alloyed and uniformly distributed in a Fe-Cr matrix. This composite structure will heal the damages due to irradiation and creep. In the second approach, iron nano-powders will be oxidized in a controlled atmosphere to form a thin shell of the oxide layer and a metal core. The metalcore/oxide-shell nano-capsules will be uniformly distributed in the Fe-Cr matrix. Tensile samples will be prepared and deformed at high temperatures. When the cracks shear the oxide-shell of the nano-capsules, the core metal will diffuse out and heal the crack by filling it. The size and distribution of the nano-capsules will be optimized for enhanced crack healing properties. The proof-of-concept of this work will attract federal and private funding because this project involves both fundamental and applied research.

Total Amount Requested: \$197,600

Resource Commitment

The University of Idaho approaches research through a sense of discovery and focuses on a broad spectrum of technologies, including materials fabrication and characterization. This proposal aligns well with the university's mission and priorities. Therefore, the required resources will be available to complete this proposed project. The research team has all the facilities on the Idaho Falls campus and CAES to accomplish the research tasks. Details of these facilities are provided in

Appendix A. The metal powders, chemical reagents, and other supplies needed for the project will be procured from the vendors as needed.

Specific Project Plan and Timeline

Background: Nuclear and other power plant components are exposed to aggressive service conditions, including high temperatures, high stresses, irradiation, and corrosion, which lead to damage accumulation and restricted service lifetime. If the structural materials used in these components can self-heal the microscopic damages induced by irradiation, creep, and other degradation mechanisms, their lifetime can be extended considerably. The self-healing approach can also improve the reliability, sustain safety, and extend the life of current or advanced reactors. *Self-healing of metallic components is considered a holy grail.*

Coatings are applied to protect the surfaces of the load-bearing structures against aggressive environments from excessive wear, corrosion, and oxidation. Stress fields induced by thermal cycling, flow conditions, vibrations, and service load variations cause cracking of the protective coatings. The lifetime of the coatings can significantly be extended if the cracks are self-healed. Self-healing polymer coatings are widely used in aqueous corrosion applications. However, self-healing ceramic coatings for high-temperature coatings are still in their early stage of development. This proposal addresses this knowledge gap. Metal core-oxide shell nano-capsules will be prepared by a simple chemical or thermal route and mixed with corrosion-resistant alloy powders. Bulk components can be manufactured via powder metallurgy route by compaction and sintering. The metal alloy–nanocapsule mixture can be cold sprayed and annealed to render a self-healing coating on the structural components.

Self-healing of the radiation-induced damage was demonstrated by atomic simulation studies at Los Alamos National Laboratory [1]. Irradiation leads to the creation of point defects such as interstitial-vacancy pairs. The interstitials diffuse to the surface, and other defect sinks, causing vacancies to condense as voids, resulting in swelling. Furthermore, radiation damage can lead to embrittlement. The radiation damage is minimized or removed by promoting the recombination of vacancy-interstitial pairs. This is achieved by having nanomaterials whose grain boundaries can bind the interstitial atoms and release them to capture vacancies adjacent to the grain boundaries. Damage site-specific precipitation of solute atoms such as Au, Mo, or W in Fe-X alloys was also reported to reduce the radiation swelling [2, 3]. Site-specific precipitation of solute atoms was observed to self-heal creep cavities of steel [4], however, only microscopic damages caused by stress, such as self-healing by shape memory effect [5], electrodeposition [6], and distribution of microcapsules containing reactive materials in a coating material [7].

Overall, the proposed research focuses on developing advanced materials and manufacturing techniques for applications of next-generation power plant components, including nuclear reactors, more specifically for molten salt reactors. The materials can also be used in the currently operating LWR nuclear reactors for enhanced safety and reliability.

Proposed Innovative Approach:

Figure 1 illustrates the proposed innovation of this project. Distribution of nano-capsules having metal-core and oxide-shell will have multiple advantages such as: i) nano-scale interfaces act as sinks for irradiation-induced point defects and annihilate them, ii) damage accumulation in the form of dislocations and movement of mobile dislocations shears the nano-capsules and exposes the metal core and facilitates site-specific precipitation of the solute metal atoms as FeB₂ at the



Figure 1. Schematic illustration of self-healing behavior of the proposed material. (a) conventional material having a crack and irradiation induced cavities and voids. (b) Composite material with uniform distribution of metal coreoxide shell nano-capsules. (c) Healing of crack by filling with the metal from the core of the broken nano-capsules, and site- specific filling of the vacancies by the metal-core when dislocations shear the nano-capsules.

irradiation-induced voids, and iii) uniform distribution of the nano-capsules helps to heal the cracks when the crack front shears the nano-capsules and releases the metal that fills the crack.



Fig. 2 *Manufacturing of mechanically alloyed Ni4W-NiW composite alloy. (a) Morphology of as-ball milled powder, (b) Heat treated powder, c) XRD of heat treated Ni4W-NiW composite.*

Previous Related Work:

The team successfully manufactured nano-composite Ni₄W - NiW powders by mechanical alloying pure elemental powders to demonstrate the site-specific precipitation of Ni₄W in the Ni-W matrix to heal the irradiation damage. 65 mol% nickel powder (3 - 7 micron, Thermo Fisher Scientific, 99.9% purity metal basis), and 35 mol% tungsten powder (1 - 5 micron, Thermo Fisher Scientific, 99.9% purity metal basis) were mixed in a milling canister to create a total of 50 grams of Ni-35 pct W powder per batch. 50 ml of hexane (C₆H₁₄) was added as a surfactant. 500 grams

of 10-mm milling balls (10:1 ball-to-powder weight ratio) were used for milling. The milling canister was sealed and inserted into a PQ-N2 Planetary Ball Mill (manufactured by Across International). Ball milling commenced for 50 hours at 300 rpm. The mechanically alloyed powders were heat treated (HT) in a high-temperature tube furnace (model H18-40HT, manufactured by Micropyretics Heaters International) for 1 hour at 800°C and then air cooled. A steady argon gas was passed through the furnace during the HT. Fig 2 (a) shows the morphology of the ball-milled Ni₄W-NiW powder composite. No significant change in the morphology was observed after heat treatment, as seen in Fig. 2 (b). The XRD pattern in Fig. 2 (c) revealed the formation of predominantly the Ni₄W-NiW composite structure.

Relevancy to HERC priorities: This proposed research addresses two priority areas specified in the RFP. These are i) Energy and ii) Human-Environment Interactions. Energy security and safety depend on the integrity of the components used in the power generation. This proposed research unveils a novel concept of self-healing of the defects in metallic components. Such a self-healing phenomenon is utilized in the organic coatings. This work extends the self-healing to inorganic materials. Successful implementation of this project will place Idaho in a unique position to lead the nation in the reliability of energy conversion. The proof-of-concept of this work will attract federal and private funding because this project involves both fundamental and applied research.

RESEARCH & DEVELOPMENT PLAN

The objectives of this project are to:

Prepare Fe-16 Cr alloy powders (referred to as FeCr alloy) by high energy ball milling of pure elemental powders in required quantities as a matrix to distribute nano-phases;

Prepare metal-core/oxide-shell nano-capsules (MCOS) by controlled oxidation of Fe nano-powders;

Prepare sintered composite test coupons of FeCr-MCOS nano-capsule composites;

Characterize the microstructures of the composite materials; and

▶ Investigate the self-healing behavior of the sintered specimens by carrying out interrupted tensile tests at room temperature and annealing the tensile tested specimens at 500 – 700 °C in an inert environment;

Task 1: Preparation of Alloy Powders

<u>Objective</u>: To prepare solid solutions of FeCr and Fe-B alloy powders as matrix materials for the preparation of composite materials.

<u>Approach</u>: High-energy ball milling will be carried out to prepare the mechanically FeCr alloy powders as the matrix material. The elemental powders of Fe, and Cr will be weighed in required quantities to obtain a composition of Fe-16Cr. The powders will be procured from Alfa Aesar with 99.8% purity and a mesh size of 150 to 200 (75 – 100 microns). All the powders will be handled inside a controlled atmosphere glove box filled with ultra-high purity argon to minimize contamination due to oxygen and moisture. The powder mixture will be mechanically alloyed to obtain solid solutions of the FeCr alloy. Mechanical alloying will be achieved by high-energy ball

milling using WC balls with a metal-to-ball ratio of 1:10 at 300 – 500 rpm for up to 100 h. Argon cover and hexane will be used to minimize the oxidation of powders during ball milling. To ensure complete alloying and formation of a single-phase solid solution structure, the ball-milled powders will be solution annealed at 1050 °C for 30 minutes in an inert environment. A second batch of alloy powder containing Fe-1%B will be mechanically alloyed to investigate the site-specific precipitation of Fe2B on the damage accumulation sites.

Deliverables: Alloyed powders of Fe-Cr and Fe-B for composite preparation in Task 3.

Task 2: Synthesis of Metal-Oxide Core-Shell Nano-capsules

<u>Objective</u>: To prepare the metal-core/oxide-shell (MCOS) nano-capsules for rendering self-healing properties

<u>Approach</u>: In this task, 60-80 nm size pure iron nanopowder will be procured from Sigma-Aldrich. These nanoparticles typically have about 2 nm thick shell of FeO. The Fe nano-powders will be thermally oxidized in a commercial purity argon atmosphere at 250 - 300 °C for a short and sufficient time to obtain an oxide layer thickness of ~10 nm. The partial pressure of oxygen (po_2) in the commercial purity of argon is about 1 Pa, based on our experience.

<u>Deliverable</u>: The MCOS nano-capsules for composite preparation in Task 3.

Task 3: Preparation of Self-Healing Composites

<u>Objective</u>: To prepare self-healing FeCr-MOCOS, and Fe-B composites using mechanically alloyed powders.

<u>Approach</u>: The matrix (FeCr) and dispersoids (MCOS) will be thoroughly mixed at different ratios (volume fraction of MCOS will be in the range of 10 - 40 %) along with organic binders and compacted at about 100 MPa to prepare cylindrical preforms of 12 mm diameter. The preforms will be sintered in an inert environment at 1000 °C for 1 - 8 h. Another batch of the mixed powders will be spark plasma sintered at 1000 °C at a pressure of 100 MPa for about 600 seconds in a vacuum using graphite dies. ASTM E8 subsize tensile samples will be machined out of the sintered cylinders. Fe-B mechanically alloyed powders will be consolidated and sintered using a similar procedure, and tensile coupons will be machined out to evaluate the self-healing behavior by site-specific precipitation of Fe₂B on the micro-cracks during tensile testing.

<u>Deliverables</u>: Tensile samples of FeCr-MCOS and Fe-B sintered composite materials for further characterization.

Task 4: Mechanical Testing and Evaluation of Self-healing:

<u>Objective</u>: To prove the self-healing behavior of the composite materials by conducting interrupted tensile testing on the FeCr-MCOS and Fe-B composites.

<u>Approach</u>: ASTM E8 sub-sized tensile specimens of FeCr-MCOS and Fe-B will be tested at different temperatures (20 - 700 °C) at 10^{-4} or 10^{-5} s⁻¹ tensile strain rates to different strain levels. The tensile tests will be interrupted after crack initiation for some specimens to investigate the crack healing behavior. A set of crack-initiated specimens will be annealed at 700 °C for 1-8 h in

an argon atmosphere, followed by water quenching and tensile testing again at room temperature. Another set of pre-strained specimens will be tensile tested without crack-healing annealing treatment, and the results will be compared.

<u>Deliverables</u>: Understanding the mechanism of crack healing behavior and improving mechanical behavior after crack healing. The cracks during the tensile testing are anticipated to be healed by diffusion of Fe from the sheared MCOS in FeCr-MCOS specimens and by filling the microcracks with Fe₂B precipitation in the Fe-B specimens. The healing process will result in higher tensile strength of the FeCr-MCOS composite material than the base FeCr alloy.

Task 5: Microstructural Characterization of the Pre and Post-Tested Specimens

<u>Objective</u>: To evaluate the microstructures of the composites and microstructural evolution during crack healing.

<u>Approach</u>: An extensive microstructural characterization will be carried out on the as-prepared and post-tested specimens using FE-SEM, XRD, Raman microscopy, and TEM analyses to evaluate crack healing behavior and corrosion resistance of the new materials developed in this project. These results will be critically analyzed to optimize the nanocapsules' size, distribution, and volume fraction required for effective crack healing and the manufacturing process parameters.

<u>Deliverable</u>: Microstructural data of the composite materials and mechanistic understanding of the crack healing behavior. The data will also be used for optimizing MCOS nano-capsule size, volume fraction (inter-particle spacing), and distribution.

Task	Description	Project timeline months											
		Q	L	Q2	2	Q3		Q4		Q5		Q6	
1	Preparation of FeCr, Fe-B alloy powder												
2.	Synthesis of Metal-Oxide Core-Shell Nano-capsules												
3	Preparation of Self-Healing Composites												
4	Mechanical Testing and Evaluation of Self-healing												
5	Microstructural Characterization of the Pre and Post-Tested Specimens												
8	Reports												

<u>Timeline</u>

Milestone 1: Preparation of FeCr and Fe-B alloy powder at the end of 1st quarter Milestone 2: Preparation of Fe-Fe_xO_y core-shell nanocapsules with desired dimensions at the end of 2nd quarter Milestone 3: Preparation of sintered coupons at the end of 3rd quarter Milestone 4: Results of crack healing tensile tests and proof of concept of crack healing at the end of 5th quarter Milestone 5: Results of microstructural and self-healing optimization at the end of 6th quarter.

8. Potential Economic Impact

This project proposes a unique concept of self-healing of metallic components. The commercially available self-healing coatings are only polymer-based. Passive films of aluminum and chromiumcontaining alloys are considered self-healing, but the proposed concept is totally different from the above. The proposed self-healing is related to healing cracks and damages due to mechanical loading. This concept will increase the lifetime of the components significantly and decrease the inspection and maintenance costs. This concept can also be extended to protective coatings. The self-healing coating market is valued at \$2.4 billion in 2023 and is expected to grow 34.2% in 2023-2028. Successful implementation of this project will have tremendous commercial potential, especially when applied to advanced manufacturing methods. The nuclear or power generation industries and other industries such as aircraft, aerospace, automobile, oil, and chemical industries will be interested in adopting this technology. Self-healing of metallic components is one of the holy grails that society is looking for.

9. Criteria for measuring success

Success will be measured based on achieving the milestones and meeting the timelines, which are given in an earlier section. An additional metric for measuring the project's success will be the submission of at least two manuscripts to refereed journals for publication. Another metric for success is the submission of a research grant proposal to a federal agency seeking additional funding to expand the proof of concept to the pilot scale. The current TRL is 1-2. The successful completion of the project will make the TRL 3 or 4. If we reach TRL 4, the project will be highly successful. Two graduate students will be supported in this project for 3 semesters and graduate with their M.S. degrees.

10. Anticipated Development Challenges/barriers

This proposed concept of self-healing metallic components by shearing the metal core-oxide shell (MCOS) nano-capsules is new and yet to be demonstrated. A similar concept has been demonstrated on polymer and ceramic materials. The major challenge of healing metallic components is keeping the metal core without getting oxidized. Typically, the oxide shell of the MCOS will be designed to prevent oxidation of the metal core, even during the processing and service conditions. However, when the MCOS is sheared, the metal core will be exposed, and the metal core may be oxidized depending on the partial pressure of oxygen in the medium. If an internal crack shears the MCOS, there will be no oxygen, so no oxidation occurs. On the other hand, surface cracks could oxidize the metal core. To overcome this issue, a larger volume fraction of MCOS nano-capsules is required closer to the surface than in the bulk. Other possible challenges

are i) the micro-cracks do not meet the MCOS nano-capsules before they grow longer, or the dislocations bow out without shearing the nano-capsules. These challenges will be addressed by optimizing the volume fraction and uniform distribution of the MCOS nano-capsules, and designing the MCOS particle size and inter-particle spacing based on the well-established Orowan's theory of dispersion strengthening. The proposed spark plasma sintering or conventional sintering techniques for manufacturing composites overcome some of the challenges faced by other advanced manufacturing methods, such as directed energy deposition or cold spray. For example, during cold spray, the MCOS nano-capsules could be sheared by the impact during the deposition. The fusion processes, such as directed energy deposition, could result in the floating of the MCOS nano-capsules by the buoyancy effect. The proposed spark-plasma sintering will result in the uniform distribution of the MCOS nano-capsules, and ensure could bonding between the MCOS and the alloy matrix.

11. Budget

Please check the attached budgets (Form D: IGEM-HERC Full Proposal Budget).

12. Budget Justification

Table below provides an itemized budget for the proposed project. For more details, please check **Form D** and **Appendix D**. The project period is July 01, 2024 – December 31, 2025 (18 months).

LINE ITEM REQUEST	JUSTIFICATION	TOTAL REQUEST
Personnel (salary and fringe)	Support for PI, Co-PI, and 2 graduate students	124,285
Equipment	-	-
Travel	-	-
Participant Support		
Other Direct Costs	Materials/supplies, publication, and tuition and health insurance for graduate students	73,304
		\$197589

The explanation of costs indicated in the budget above is as follows:

1. Personnel Costs (salary and fringe): PI Raja and co-PI Charit will spend 1 summer month each working on the project. Two graduate students will execute the project tasks for 3 semesters.

The salary and fringe bases are as shown in the attached Form D worksheet. An anticipated fringe rate is applied to this budget. Per the university guidance on new proposals, anticipated rates as 'estimated fringe rates' for projects that will begin 7/1/24 and later are applied. FY25 anticipated fringe rates are as follows: Faculty: 31.7%; Staff: 40.1%; Students: 2.0%; Temporary:10.1%. Current FY24 fringe rates available at: http://www.uidaho.edu/research/faculty/resources/fringe-rates are: Faculty: 31%; Staff: 41.3%; Students: 2.5%; Temporary: 8.3%.

2. Other direct costs include supplies cost of \$30,000 (itemized list in Form D), publication costs of \$5,180, and graduate student tuition (\$31644) and health insurance (\$6480) for 2 students, 3 semesters.

13. Project Management

PI Raja will serve as the overall project director and will be the lead responsible for tasks 1-4. Co-PI Charit will lead the experimental efforts on the Task-5 and closely work with the PI on other tasks. Two graduate students will conduct the project activities, with the PI and co-PI serving as the major professor/advisor for one student each. The research group will hold biweekly meetings to review the activities and analyze the data. In addition, both PI and co-PI will be available and accessible to both the graduate students for consultation and discussion as needed. PI and co-PI have collaborated extensively on several research projects and developed a smooth *modus operandi* for successful accomplishment of project objectives.

<u>Process for making decisions on scientific/technical direction</u>: The project team will function collaboratively, with the overall direction and approach developed through consensus building and discussions. The investigators who have the major responsibility for those tasks will direct the detailed execution of the tasks, as described later in this document. The project team will hold regular reviews of the investigation results to interpret them and make decisions regarding possible refinement/revision of the approach.

<u>Publications</u>: The research results and outcomes will be disseminated to the public through presentations at the regional and national meetings of technical societies (American Nuclear Society - ANS, American Institute of Chemical Engineers - AIChE, or The Minerals, Metals, and Materials Society - TMS), and refereed publications in the peer-reviewed technical journals. Only those individuals who have contributed significantly to the article will be listed as authors of the publication. The support of HERC - Idaho State Board of Education will be acknowledged in all the presentations of oral and written dissemination. In addition, quarterly and annual reports of the project's progress will be submitted to the HERC office.

<u>Intellectual Property Issues</u>: All intellectual property issues will be resolved according to the existing policies of both institutions. The university's intellectual property policy is codified in the Faculty-Staff Handbook and is available at <u>http://www.webpages.uidaho.edu/fsh/5400.html</u>. The Office of Technology Transfer (http://www.uidaho.edu/research/about/ott) at the University of Idaho provides support and manages the intellectual property issues related to the discoveries at the university. The university has also established a new expanded intellectual property policy recently, which has eased the process for partnership with entities external to the university.

14. Additional institutional and other sector support

The institutional support for the project is manifest through the availability of the lab space, utilities, and project administration support. Due to the nature of the project (proof of concept) and the low TRL of 2, an industrial partner is not currently involved in the project. However, potential industrial collaborations are identified as described below.

15. Future funding

Several avenues are identified for securing funding for future research and technology development. These include federal funding agencies and industrial partnerships. Federal funding agencies/programs that sponsor research include the Department of Energy, NSF, and Department of Defense units such as DARPA and AFSOR. We collaborate with small businesses to submit SBIR/STTR proposals. Currently, we are collaborating with a small business GenNext Materials & Technologies, LLC (GMT), on an STTR Phase I project funded by the U.S. Department of Energy (USDOE). Our project "3D Printing of Functionally Graded in-situ Sacrificial Anode Claddings for Enhanced Corrosion and Irradiation Resistance in MSR Applications" was approved for funding on 02/22/2024. We will continue collaborating with this small business (a support letter is included in Appendix D) and submit SBIR/STTR proposals. The proof of concept of this proposal will help explore further funding opportunities to expand to the next TRL level and ultimately reach commercialization by teaming up with a large-scale manufacturing industry.

References

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Form C: IGEM-HERC Institutional & Other Sector Support

(add additional pages as necessary)

A. LIST INSTITUTIONAL/ OTHER SECTOR DOLLARS	Amount
(Source & Description)	

B. FACULTY/ STAFF POSITIONS	Amount
(Description)	

C. CAPITAL EQUIPMENT	Amount
(Description)	

D. FACILITIES & INSTRUMENTATION	Amount
(Description)	

FORM D: I	GEM-HER	C Full Propos	al Budget She	et					
elect one):	Proof of C	Concept							
ast Name:	Krishnan S	S Raja							
ject Title:	Self-healin	g Composites f	or Aggressive	Environments					
Milestone	descriptio	Enter mileston	e number and	/or brief desc	ription he	ere			
Insert more r	ows in each	section, as needed	Copy/paste cell	formulas, as nee	eded.	See cell no	otes for additiona	l information.	
Do not remov	/e or hide rov	vs.	Shaded areas ha	ave preset formu	ılas.				
Personnel									
Name	FTE (opt)	Months	Base Salary	Salary Reque	Fringe Ra	Other Ber	Fringe Reques	Total	
Krishnan S I	Raja	1	\$121,134.00	\$10,094.50	0.317		\$3,199.96	\$13,294.46	
Indrajit Char	it	1	\$144,190.80	\$12,015.90	0.317		\$3,809.04	\$15,824.94	
Graduate St	udent-1	18	\$31,100.00	\$46,650.00	0.02		\$933.00	\$47,583.00	
Graduate St	udent-2	18	\$31,100.00	\$46,650.00	0.02		\$933.00	\$47,583.00	
		1	\$0.00	\$0.00			\$0.00	\$0.00	
		1	\$0.00	\$0.00			\$0.00	\$0.00	
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Item	L Inite	Cost						Total	
Rom	OTINO	0001						10101	
Materials/									
Supplies	1	\$30,000.00						\$30,000.00	
Publication									
Charges	2	\$2,590.00						\$5,180.00	
Consultants	1							\$0.00	
0									
Computer								¢0.00	
Subcontract	1							\$0.00	
	4							\$0.00	
Subcontract								\$0.00	
s 2								\$0.00	
Other	1							\$0.00	
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students 3									
semesters	6	\$5 274 00						\$31.644.00	
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				T <u>O</u>	TAL <u>DIRE</u>	ECT <u>COS</u>	T REQUEST	\$197 <u>,589.40</u>	
								12	Page

Appendix A

Facilities and Equipment

Dr. Raja has the following facilities available in his laboratory:

(1) <u>Material preparation</u>: A planetary ball mill for preparing the FeCr and Fe-B matrix as shown in Fig. 3, A controlled atmosphere glove box capable of maintaining < 0.2 ppm oxygen and moisture



Fig. 3. Images of the planetary ball mill for preparation of the alloy matrix.



Fig. 4 Controlled atmosphere glove box that can maintain oxygen and moisture less than 2 ppm.



Fig. 54 Confocal Raman Microscope (Horiba, XPloRA Plus) with a Linkam Hot stage. Raman spectra can be obtained in- situ while heating the samples to 1100 °C

(mBraun, Model: LabStar) (Fig. 4), several potentiostats (Gamry Instruments, Models: Reference 600 and Interface 1000, Princeton Applied Research (AMETEK), Model: VersaSTAT MC (4channels), and CH Instruments: Model 400C Electrochemical quartz microbalance). Seven experiments can be carried out at the same using the potentiostats.

(2) <u>Material Characterization</u>: **Confocal Raman Microscope, Make: HORIBA, Model: XPloRA** Plus with LINKAM Hot stage capable of heating molten salts to 1100 °C in an argon atmosphere and conducting in-situ microscopy (Fig. 5). Three laser wavelengths 532, 638, and 785 nm each 100 mW, motorized stage capable of mapping the sample (3) <u>Spectroscopic Facility</u>: Raman spectrometer (i-Raman with 532 nm laser, Gamry Instruments) and in-situ UV-Vis spectroscope (Spectro 115E, 350-1050 nm, Gamry Instruments) for in-situ spectroscopic investigation during electrochemical testing. (4) <u>Thermal treatment facilities</u>: Two controlled atmosphere furnaces with ramping capabilities with a maximum temperature of 1300 °C (Nabertherm, Model: RD/30/200/13) and 1700 °C (SentroTech, Model STT-1700C), Vacuum Oven: (Max: 250 °C, 1 cu.ft., V0 – 0.1 MPa Vacuum reading, Jeio Tech Co., Model: OV-11-120). (5) <u>Other experimental testing facilities</u>: Microbalance (Radwag USA, Model: XA110/2X), and Millipore water purification system capable of supplying high purity water with 18.2 Megaohm-cm resistivity, several non-destructive testing capabilities including ultrasonic flaw detector (GE Inspection Technologies, Model: USM Go), Eddy current tester, magnetic flux detector. An ICP-MS (Shimadzu ICPMS 2030) is available in the department with unlimited access for analyzing liquid and gaseous samples.

Dr. Charit has a 5982 model Instron universal tester for carrying out high temperature tensile tests (fitted with Epsilon Technologies 3549 high temperature extensometer) and an Applied Test Systems 2335 lever arm (20:1) creep tester for performing high temperature creep tests. Both instruments are fitted with furnaces capable of reaching 1000 °C. In addition, a LECO LM-100 microhardness tester is available for doing Vickers microhardness testing. Dr. Charit's laboratory also has a Netzsch Simultaneous Thermal Analyzer (capable of simultaneously performing three thermal analysis techniques, differential scanning calorimetry (DSC), differential thermal analysis (DTA) and thermogravimetric analysis (TGA) up to 1500°C, high temperature furnaces including one MHI furnace capable of going up to 1700oC, ball milling equipment (SPEX Mill 8000M), SPEX 2380 bench press, glove box, a complete TEM sample preparation kit (Gatan Disc Punch, Fischione Disc Grinder and Twin-Jet Polisher), and a metallography facility (Buehler precision saw, an Allied HighTech TwinPrep grinder/polisher, a Pace Technologies hot mounting press, a vibratory polisher and an Amscope optical microscope).

The university investigators have access to various other advanced characterization instruments located at the Center for Advanced Energy Studies (CAES) such as Microstructure and Characterization Suite (MaCS), and Advanced Materials Laboratory (AML) The MaCS is a state-of-the-art microstructural characterization facility housed inside the having the following instruments: Scanning Transmission Electron Microscope (STEM) - ThermoFisher Spectra 300 - monochromated, double-corrected, 30 - 300kV; Scanning Transmission Electron Microscope - FEI Tecnai TF30-FEG STwin TEM with EDS, EELS (GIF), EFTEM & TopSpin; Dual Beam Focused Ion Beam Microscope (FIB) - FEI QUANTA 3D FEG with EDS & EBSD; Scanning Electron Microscope (SEM) - JEOL JSM 6610LV with EDS, EBSD, & CL; Local Electrode Atom Probe (LEAP) - Cameca LEAP 4000X HR; Nano Indenter Atomic Force Microscope - Hysitron TI950 TriboIndenter; X-Ray Diffractometer (XRD) - Rigaku SmartLab.

Appendix B: Biographical Sketches

IDENTIFYING INFORMATION:

NAME: Raja, Krishnan Selva

ORCID iD: https://orcid.org/0000-0003-4746-2272

POSITION TITLE: Professor of Nuclear Engineering

PRIMARY ORGANIZATION AND LOCATION: University of Idaho, Idaho Falls, Idaho, United States

Professional P	reparation:				
ORGANIZ	ATION AND LOCATION	DEGREE	RECEIPT	FIELD OF STUDY	
		(if applicable)	DATE		
Indian Institute of Technology, Chennai, Not Applicable, N/A, India		PHD	07/1993	Welding and Stress Corrosion CrackingMetallurgical Engineering	
Indian Instite Not Applical	ute of Technology, Chennai, ple, N/A, India	MENG	02/1988	Industrial Metallurgy	
College of Engineering, Guindy (Anna University), Chennai, Not Applicable, N/A, India		BENG	05/1986	Mechanical Engineering	
Appointments	and Positions		•	'	
2023 - present	Professor of Nuclear Engine States	ering, Universit	y of Idaho, Io	daho Falls, Idaho, United	
2021 - 2022 Professor of Materials Engineering, University of Idaho, Moscow, Idaho, Unite States					
2016 - 2021 Associate Professor, Materials Engineering, University of Idaho, Moscow, Ida United States					
2011 - 2016	11 - 2016 Assistant Professor of Materials Engineering, University of Idaho, Moscow,Idaho, United States				
2001 - 2011	Research Faculty - Chemical Reno, Nevada, United States	l and Materials : s	Engineering,	University of Nevada,	

1997 - 2000 Research Associate, Tohoku University, Sendai, Not Applicable, N/A, Japan

1993 - 1997 Research Executive Engineer, Larsen & Toubro Ltd., Mumbai, Not Applicable, N/A, India

Products

Products Most Closely Related to the Proposed Project

- Thuneman T, Raja K, Charit I. Room Temperature Corrosion Behavior of Selective Laser Melting (SLM)-Processed Ni-Fe Superalloy (Inconel 718) in 3.5% NaCl Solution at Different pH Conditions: Role of Microstructures. Crystals. 2024 January 18; 14(1):89-. Available from: https://www.mdpi.com/2073-4352/14/1/89 DOI: 10.3390/cryst14010089
- 2. Singla Y, Miller J, Raja K, Maughan M. Toward single crystal nickel fabrication using WAAM
- A first report. Journal of Materials Research and Technology. 2023 November; 27:4801-4804.
 Available from: https://linkinghub.elsevier.com/retrieve/pii/S2238785423027898 DOI: 10.1016/j.jmrt.2023.11.016
- Naskar A, Bhattacharyya M, Raja K, Charit I, Darsell J, Jana S. Room temperature corrosion behaviour of plastically deformed AISI 304 stainless steel by friction stir welding in neutral and acidified chloride solutions. Corrosion Engineering, Science and Technology. 2022 July 30; 57(7):599-612. Available from: https://journals.sagepub.com/doi/full/10.1080/1478422X.2022.2105682 DOI: 10.1080/1478422X.2022.2105682
- Bhattacharyya M, Gnaupel-Herold T, Raja K, Darsell J, Jana S, Charit I. Evaluation of residual stresses in isothermal friction stir welded 304L stainless steel plates. Materials Science and Engineering: A. 2021 October; 826:141982-. Available from: https://linkinghub.elsevier.com/retrieve/pii/S092150932101248X DOI: 10.1016/j.msea.2021.141982
- Naskar A, Bhattacharyya M, Raja K, Charit I, Darsell J, Jana S. Pitting behavior of friction stir repair-welded 304L stainless steel in 3.5% NaCl solution at room temperature: role of grain and defect structures. SN Applied Sciences. 2020 December 06; 2(12):-. Available from: http://link.springer.com/10.1007/s42452-020-03935-0 DOI: 10.1007/s42452-020-03935-0

Other Significant Products, Whether or Not Related to the Proposed Project

- Vaidya T, Stanford J, Rooyen N, Raja K, Utgikar V, Sabharwall P. Capture of Volatile Organic Iodine Species Using Mordenites. Journal of Nuclear Fuel Cycle and Waste Technology(JNFCWT). 2023 June 30; 21(2):205-224. Available from: https://www.jnfcwt.or.kr/journal/article.php?code=86875 DOI: 10.7733/jnfcwt.2023.016
- Dhabarde N, Ferrer A, Tembo P, Raja K, Subramanian V. Chalcogenide-Based Complex Transition Metal Electrocatalyst for Hydrogen Peroxide Production. Journal of The Electrochemical Society. 2023 January 16; 170(1):016506-. Available from: https://iopscience.iop.org/article/10.1149/1945-7111/acafa5 DOI: 10.1149/1945-7111/acafa5

- Goettsche H, Raja K, Sabharwall P, Utgikar V. Treatment of Off-Gas Emissions: Kinetics of Silver Mordenite Catalyzed Methyl Iodide Decomposition. Chemical Engineering Journal Advances. 2022 May; 10:100290-. Available from: https://linkinghub.elsevier.com/retrieve/pii/S2666821122000515 DOI: 10.1016/j.ceja.2022.100290
- Dhabarde N, Carrillo-Ceja O, Tian S, Xiong G, Raja K, Subramanian V. Bismuth Vanadate Encapsulated with Reduced Graphene Oxide: A Nanocomposite for Optimized Photocatalytic Hydrogen Peroxide Generation. The Journal of Physical Chemistry C. 2021 October 26; 125(43):23669-23679. Available from: https://pubs.acs.org/doi/10.1021/acs.jpcc.1c05315 DOI: 10.1021/acs.jpcc.1c05315
- Shrestha N, Raja K, Utgikar V. Mg-RE Alloy Anode Materials for Mg-Air Battery Application. Journal of The Electrochemical Society. 2019 September 17; 166(14):A3139-A3153. Available from: https://iopscience.iop.org/article/10.1149/2.0131914jes DOI: 10.1149/2.0131914jes

Synergistic Activities

- 1. Licensed Professional Engineer (Metallurgical and Materials Engineering in the state of Idaho (P16254);
- 2. NACE Certified Materials Selection and Design Specialist (Certification # 25861) 3.

Editorial Board Member of Metallurgical and Materials Transactions A, Springer

Certification:

When the individual signs the certification on behalf of themselves, they are certifying that the information is current, accurate, and complete. This includes, but is not limited to, information related to domestic and foreign appointments and positions. Misrepresentations and/or omissions may be subject to prosecution and liability pursuant to, but not limited to, 18 U.S.C. §§ 287, 1001, 1031 and 31 U.S.C. §§ 3729-3733 and 3802.

Certified by Raja, Krishnan Selva in SciENcv on 2024-03-04 17:21:22

IDENTIFYING INFORMATION:

NAME: Charit, Indrajit

ORCID iD: <u>https://orcid.org/0000-0002-3854-2900</u>

POSITION TITLE: Professor and Department Chair

<u>PRIMARY ORGANIZATION AND LOCATION</u>: Department of Nuclear Engineering and Industrial Management, Idaho Falls, Idaho, United States

Professional 1	Preparation:				
ORG	ANIZATION AND LOCATION	DEGREE	RECEIPT	FIELD OF	
		(if applicable)	DATE	STUDY	
Missouri Un	iversity of Science and Technology,	РНО	05/2004	Metallurgical	
Rolla, Misso	uri, United States	1112	00/2001	Engineering	
Indian Instit	rute of Science, Bangalore, Not	MENG	01/2000	Metallurov	
Applicable,	N/A, India		01/2000	wietuniargy	
Indian Ir	nstitute of Engineering Science and	BENG	06/1997	Metallurgical	
Technology	, Shibpur, Not Applicable, N/A, India	DENC	00/1777	Engineering	
Appointment	<u>s and Positions</u>				
2021 - present	Professor and Department Chair, D	epartment of Nu	uclear Engine	eering and	
Industrial Ma	nagement, University of Idaho, Idaho F	alls, Idaho, Unit	ed States		
2020 - present	Affiliate Professor, Department of Me	chanical Engine	ering, Unive	rsity of Idaho,	
	Moscow, Idaho, United States				
2010 - present	Affiliate, Center for Advanced Energy S	tudies, Idaho Fa	alls, Idaho, U	nited States	
2023 - 2023	Interim Center Executive Officer, Uni	versity of Idaho	- Idaho Falls	Center, Idaho	
	Falls, Idaho, United States				
2020 - 2020	Professor and Director, Materials Scie	nce and Engine	ering Progra	m, University	
	of Idaho, Moscow, Idaho, United Stat	es			
2019 - 2020	2019 - 2020 Professor, Department of Chemical and Materials Engineering, University of				
	Idaho, Moscow, Idaho, United States				
2015 - 2020 I	Director of Graduate Studies, Departme	nt of Chemical a	nd Materials	Engineering,	
	University of Idaho, Moscow, Idaho,	United States			

2013 - 2019	Associate Professor, Department of Chemical and Materials Engineering, University of Idaho, Moscow, Idaho, United States
2010 - 2013	Assistant Professor, Department of Chemical and Materials Engineering, University of Idaho, Moscow, Idaho, United States
2008 - 2008	Visiting Faculty, Materials and Fuels Complex, Idaho National Laboratory, Idaho Falls, Idaho, United States
2007 - 2010	Assistant Professor, Department of Materials Science and Engineering, University of Idaho, Moscow, Idaho, United States
2005 - 2007	Postdoctoral Research Associate, Department of Nuclear Engineering, North Carolina State University, Raleigh, North Carolina, United States
2004 - 2004	Postdoctoral Fellow, Department of Materials Science and Engineering, Missouri University of Science and Technology, Rolla, Missouri, United States
2000 - 2004	Graduate Research Assistant, Department of Metallurgical Engineering, Missouri University of Science and Technology, Rolla, Missouri, United States
2000 - 2000	Project Engineer, TVS Suzuki Limited, Hosur, Not Applicable, N/A, India
1997 - 1998	Quality Control Engineer, Electrosteel Castings Limited , Khardah, Not Applicable, N/A, India

Products

Products Most Closely Related to the Proposed Project

- Bhattacharyya M, Kundu A, Raja KS, Charit I, Darsell JT, Jana S. Microstructure-Property Correlations for Temperature-Controlled Friction Stir Welding of 304L SS Plates. Materials Science and Engineering A. 2021; 804(21):140635. Available from: https://doi.org/10.1016/j.msea.2020.140635
- Ghayoor M, Mirzababaei S, Sittiho A, Charit I, Paul BK, Pasebani S. Thermal Stability of Additively Manufactured Austenitic 304L ODS Alloy. Journal of Materials Science and Technology. 2021; 83:208-218. Available from: DOI: 10.1016/j.jmst.2020.12.033
- Goel S, Sittiho A, Klement U, Joshi S, Charit I. Effect of Post-Treatments under Hot Isostatic Pressure on Microstructural Characteristics of EBM-built Alloy 718. Additive Manufacturing. 2019; 28:727-737. Available from: https://doi.org/10.1016/j.addma.2019.06.002
- Brubaker N, Ali H, Dhakal S, van Rooyen N, Jaster M, Charit I, Jaques B, Maughan MR. Investigating Microstructure and Properties of 316L Stainless Steel Produced by Wire-Fed Laser Metal Deposition. Journal of Materials Engineering and Performance. 2022; 31:3508-3519. Available from: https://doi.org/10.1007/s11665-021-06477-7
- Roberts D, Zhang Y, Charit I, Zhang J. A Comparative Study of Microstructure and High Temperature Mechanical Properties of 15-5 PH Stainless Steel Processed via Additive Manufacturing and Traditional Manufacturing. Progress in Additive Manufacturing. 2018; 3(3):183-190. Available from: https://doi.org/10.1007/s40964-018-0051-5

Other Significant Products, Whether or Not Related to the Proposed Project

- 1. Khanal R, Ayers N, Jerred N, Benson MT, Mariani RD, Charit I, Choudhury S. Role of Zr in Lanthanides-Dopants Interactions within Uranium-Based Metallic Fuels. Nuclear Materials and Energy. 2021; 26:100912. Available from: https://doi.org/10.1016/j.nme.2021.100912
- Jerred ND. Nd, SbNd, and Sb3Nd4, and their interactions with the cladding alloy HT9. Journal of Nuclear Materials. 2020; 541:152387. Available from: https://doi.org/10.1016/j.jnucmat.2020.152387
- 3. Jerred N, Charit I, Zirker L, Cole J. Pressure Resistance Welding of MA-957 to HT-9 for Advanced Reactor Applications. Journal of Nuclear Materials. 2018; 508:265-277. Available from: https://doi.org/10.1016/j.jnucmat.2018.05.046
- 4. Charit I, Mishra RS. Effect of Friction Stir Processed Microstructure on Tensile Properties of an Al-Zn-Mg-Sc Alloy upon Subsequent Aging Heat Treatment. Journal of Materials Science and Technology. 2018; 34:214-218. Available from: https://doi.org/10.1016/j.jmst.2017.10.021
- 5. Shrestha T, Basirat M, Alsagabi S, Sittiho A, Charit I, Potirniche GP. Creep Rupture Behavior of Welded Grade 91 Steel. Materials Science and Engineering A. 2016; 669:75-86. Available from: https://doi.org/10.1016/j.msea.2016.05.065

Synergistic Activities

- 1. Key Reader, Metallurgical and Materials Transactions A
- 2. JOM Advisor, TMS Nuclear Materials Committee (from 2017–2019) Helped in developing the program agenda for the nuclear materials activities concerning symposia and publications.
- 3. Co-organizer for the symposia, "Additive Manufacturing for Energy Applications," held in TMS 2019, 2020, 2021 and 2022 Conferences
- 4. Proposal Reviewer for the DOE Office of Nuclear Energy, US Department of Energy; National Science Foundation, and DOE Office of Science.
- 5. Professional Engineer (P.E.) licensed in Idaho (# P15773)

Certification:

When the individual signs the certification on behalf of themselves, they are certifying that the information is current, accurate, and complete. This includes, but is not limited to, information related to domestic and foreign appointments and positions. Misrepresentations and/or omissions may be subject to prosecution and liability pursuant to, but not limited to, 18 U.S.C. §§ 287, 1001, 1031 and 31 U.S.C. §§ 3729-3733 and 3802.

Certified by Charit, Indrajit in SciENcv on 2024-02-22 15:05:24

Appendix C

None

Appendix D

Letter of Support



GENNEXT MATERIALS & TECHNOLOGIES LLC

March 10, 2024

John Thomas HERC Program Manager Idaho State Board of Education 650 West State Street, 3rd Floor Boise, ID 83702

Subject: Letter of support for University of Idaho's HERC project entitled, "Self-healing Composites for Aggressive Environments."

Dear Mr. John Thomas and Members of the Higher Education Research Council:

GenNext Materials & Technologies, LLC (GMT), is currently collaborating with Drs. Raja and Charit on an STTR Phase I project funded by the U.S. Department of Energy (USDOE). Our project "3D Printing of Functionally Graded in-situ Sacrificial Anode Claddings for Enhanced Corrosion and Irradiation Resistance in MSR Applications" was approved on 02/2024. We plan to explore the self-healing behavior of <u>high-pressure cold spray technology</u> as an additive manufacturing process of depositing a thicker protective coating through our collaborative effort.

GMT understands that Drs. Raja and Charit are submitting a proposal titled "Self-healing Composites for Aggressive Environments" using an entirely different approach that they have developed from the University of Idaho (UofI).

If this new University of Idaho proposal is funded, GMT will be very interested in exploring possible collaborations on the UofI new approach and how it differs from the earlier approach.

We will be delighted to help with developing appropriate proposals, through the SBIR/STTR Phase I or Phase II platforms in consultation with our current DOE Program Manager or with other offices related to this overall topical area.

We at GMT, wish the UofI team the very best with their submission.

Sincerely

Respectfully,

S.Vaidyanathan

Co-owner Vaidyanathan Subramanian GenNext Materials & Technologies, LLC (GMT), Reno, Nevada <u>rsvgmt@gmail.com</u> <u>gennextmaterials@gmail.com</u>

Page 1 of 1

FORM D: IGEM-HERC Full Proposal Budget Sheet

Track (select one):Proof of ConceptPI First & Last Name:Krishnan S RajaProject Title:Self-healing Composites for Aggressive EnvironmentsMilestone description (if applicabl Enter milestone number and/or brief descriptio

Insert more rows in each section, as needed. Do not remove or hide rows. Copy/paste cell formulas, as needed. Shaded areas have preset formulas.

FTE (opt)	Months	Base	Salary	Salary Request
		1	\$121,134.00	\$10,094.50
		1	\$144,190.80	\$12,015.90
	1	8	\$31,100.00	\$46,650.00
	1	8	\$31,100.00	\$46,650.00
		1	\$0.00	\$0.00
		1	\$0.00	\$0.00
	FTE (opt)	FTE (opt) Months	FTE (opt) Months Base 3 1 1 18 18 18 18 1 1	FTE (opt) Months Base Salary 1 \$121,134.00 1 \$144,190.80 18 \$31,100.00 18 \$31,100.00 1 \$0.00 1 \$0.00

Equipment		
Item Description	Units Unit Cost	
	1	
	1	
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	1	
	1	
	1	
Travel		

Tentative Date(s)	# persons Total days	Transit cost/ person Lodging/ day

Participant Support	
Description	# persons Cost/ Stipend

Other Direct Costs				
Item	Units		Cost	
Materials/ Supplies		1	\$30,000.00	
Publication Charges		2	\$2,590.00	

Consultants (add consultant		
travel here)	1	
Computer Services	1	
Subcontract 1	1	
Subcontracts 2		
Other (list specifics if over		
\$1,000)	1	
In-state tuition for 2 grad		
students, 3 semesters	6	\$5,274.00
Health insurance for 2 grad		
students, 3 semesters	6	\$1,080.00
Other	1	

Chemicals for iron-iron oxide nanocapsules	\$5,035.00	1
Crucibles	\$800.00	4
Iron nanopowder, (Skyspring nanomaterials, Houston) 100 g	\$375.00	10
Additive manufacturing supplies	\$1,499.00	1
Nickel and tunsten powders	\$2,400.00	1
Bond strength fixtures	\$2,800.00	1
Metallographic supplies	\$1,250.00	1
Sample machining	\$5,000.00	1
Gas	\$454.00	4
SEM, TEM, XRD analyses	\$65.00	50

sum

n here

See cell notes for additional information.

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