MEMORANDUM OF UNDERSTANDING

BETWEEN



INSTITUTE FOR PLASMA RESEARCH

Gandhinagar - 382428 (Gujarat)

AND



INTERNATIONAL ADVANCED RESEARCH CEN-TRE FOR POWDER METALLURGY AND NEW MATERIALS (ARCI), HYDERABAD

FOR

Collaboration on

DEVELOPMENT OF TUNGSTEN FIBER REINFORCED TUNGSTEN COMPO-SITE FOR PLASMA FACING COMPONENT

IPR/PTTS/ENQ/20-21/55

FEBRUARY 2021

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PREAMBLE:

This <u>24th</u> day of <u>February</u> in the year Two Thousand and Twenty-One, a Memorandum of Understanding (hereinafter referred to as 'MoU') is entered into by and between the **Institute for Plasma Research (IPR)**, (A Grant-in-Aid Institute of Department of Atomic Energy, Govt. of India), having its registered office at Bhat, Near Indira Bridge, Gandhinagar – 382428 (Gujarat) on ONE PART;

AND

International Advanced Research Centre for Powder Metallurgy, and New Materials (ARCI), An Autonomous R & D Centre of Department of Science and Technology, Government of India, Balapur P. O, Hyderabad, PIN-500005, on the OTHER PART; on the terms and conditions hereafter contained in this MOU.

AND WHEREAS, this MoU is a task specific MoU executed by and between the Institute for Plasma Research (IPR) and International Advanced Research Centre for Powder Metallurgy and New Materials (ARCI) for the purpose of collaboration in the field of material development for fusion application. Hereinafter both IPR and ARCI will be collectively referred to as 'PARTIES' and individually referred to as PARTY;

WHEREAS IPR is engaged in R&D activities in plasma science and technology, plasma fusion application related to tokamaks, design and development of plasma processing equipment for societal and industrial application. One such area includes development of plasma facing components for ITER like tokamak devices;

WHEREAS International Advanced Research Centre for Powder Metallurgy & New Materials (ARCI) is an autonomous body and a state-of-the-art facility for research and development in the field of advanced materials and associated processing technologies, which has been established under the aegis of the Government of India's Department of Science & Technology. Its major thrust areas of focus are nanomaterials, surface engineering, ceramics, fuel cells, laser processing of materials, solar energy materials and automotive energy materials. Having identified Powder Metallurgy (P/M) as one of the areas for

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initial concerted effort, ARCI has been judiciously building up facilities to complement and enhance existing national capabilities in the field of P/M.

WHEREAS ARCI and IPR jointly discussed areas of mutual interest and observed that the strengths of both the organizations could be utilized for collaboration in the field of material development for plasma facing components (PFCs) application.

WHEREAS both the PARTIES collectively decided to enter into a Memorandum of Understanding (MoU) for jointly working on collaborative areas of mutual interest related to development of tungsten fiber reinforced tungsten composite material for plasma facing components (PFCs) application;

Both the PARTIES hereby agree to this MoU as follows:

1.0 OBJECTIVE OF THE MOU:

The objective of the MoU is to develop tungsten fiber reinforced tungsten composites (Wf/W) having fracture toughness at least two times that of the pure bulk tungsten material with optimized volume fraction of the tungsten fiber using a powder metallurgical process for use as plasma facing component (PFC). The fiber will be stacked in the form of mesh as alternate layers between tungsten powders in a graphite die followed by spark plasma sintering to achieve the desirable microstructure and mechanical properties.

The types of Wf-W composites to be developed are as follows:

- (a) Type 1: Potassium (K) doped tungsten fiber composite;
- (b) Type 2: Potassium (K) doped tungsten fiber composite with engineered interface (coated fibres).



Fig 1: Schematic diagram for Wf-W composite material

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2.0 METHODOLOGY AND SCOPE OF WORK:

Both IPR and ARCI will jointly carryout research and developmental activities of the topics mentioned in the objective of this MoU in two phases based on the respective scope of work as listed below.

2.1 PHASE 1: Feasibility study, process qualification and optimization

2.1.1 Responsibility of ARCI in Phase 1:

- 1. Raw materials to be procured.
- 2. Feasibility study to be carried out on coupon samples for tungsten fiber reinforced tungsten composite for Type-1 and Type-2 as mentioned in objective.
- 3. Optimization of the process parameters using spark plasma sintering (SPS) (as per section 2.4) for fabricating tungsten fiber composite as well as coating technique for engineered interface coating.
- Consolidated samples will be characterized for its basic properties like density, grain size, micro hardness, microstructural analysis, elemental analysis, thermalconductivity
- Deliverables for Phase-1 will be coupon samples and detailed project report mentioning process studies and material properties with relevant data files (section 3.1).

If the Phase-1 is successfully completed and the results are as per the requirement in Table 3.2.1 then only Phase-2 will be executed. Otherwise, MoU will be closed and the payment will be done as per agreed terms in section titled "Financial Agreements" (Article 5.2) in the present MoU. The decision of IPR with regard to the test results matching their required specification will be final and binding.

2.1.2 Responsibility of IPR in Phase 1:

- a) IPR shall extend all possible co-operation to ARCI in carrying out the MoU work and the technical requirements.
- b) IPR shall undertake to effect payments to ARCI as per agreed terms in section titled "Financial Agreements" (Article 5.2) in the MoU.

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2.2 PHASE 2: Fabrication and testing of Wf-W composite material as per desired size and properties

2.2.1 Responsibility of ARCI in Phase 2:

After feasibility studies and optimizing parameters, the Wf-W composite samples will be fabricated as per desired size, and shape as mentioned in 3.2. Consolidated samples will be characterized for its basic properties like density, grain size, micro hardness, microstructural analysis, elemental analysis, thermal conductivity, tensile, flexural and fracture toughness properties as mentioned in Table 3.2.1. Thermal fatigue test of Wf-W composite sample will be done at IPR for its performance check. IPR for Deliverables for Phase-2 (as mentioned in 3.2) will be detailed project report (both hard and soft copy) mentioning process studies and material properties with relevant data files and deliver 3 samples each of Type-1 and Type-2 as mentioned in Section 3.2.

2.2.2 Responsibility of IPR in Phase 2:

- a) IPR shall undertake the responsibilities of thermal fatigue testing of Wf-W composite samples.
- b) IPR shall undertake responsibilities of mechanical testing of Wf-W composite sub-sized samples using Gleeble-3800 System at IPR.
- c) IPR shall undertake to effect payments to ARCI as per agreed terms in section titled "Financial Agreements" in the present MoU.

2.3 Process approach

Concept of Wf/W composite material development: A basic strategy to achieve pseudoductility is the incorporation of new ductile matrices and fibers, which needs extensive development and validation. To overcome brittleness issues when using W, a W-fiber enhanced W-composite material incorporating extrinsic toughening mechanisms can be used. This composite approach enables energy dissipation and thus stress peaks at the crack tips can be reduced and cracks can be stopped. Another option is a composite laminate made of commercially available raw materials. In general, even in the brittle regime, this material allows certain tolerance towards cracking and damage. Even when a crack has been initiated inside the material, the energy dissipation mechanism allows further load to be put towards the component until a stage when the overall material fails.

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2.4 Work elements

The work elements of the project are to be carried out in two phases. The first phase involves a detailed assessment of consolidation of tungsten matrix reinforced with bare tungsten fiber with a single objective of achieving compacts with a best possible density. This involves an extensive optimization studies covering a wide-spectrum of activities in assessing the effects of parameters such as sintering temperature and applied load in the 100 kN SPS equipment. Besides, the volume fraction of fibres is to be optimized. Each of the parameters is to be addressed in a stage-wise manner as given below:

- Effect of temperature 3 levels
- Effect of load 3 levels

The magnetron coating technique will also be optimized on the following parameters:

- Understanding deposition rate of individual coating material
- Effect of oxygen flow on coating properties

• Effect of substrate temperature on adhesion strength of individual coatings The sintered compact will be assessed through the following characterization techniques:

- a) Density
- b) Grain size
- c) Micro-structural analysis by using optical microscopy, SEM, EDAX, XRD
- d) Vickers micro-hardness
- e) Thermal conductivity
- f) Mechanical properties (tensile and flexural strength)
- g) Elemental Analysis

Based on the results obtained from the above studies, the best possible combinations of parameters will be chosen for further evaluation during Phase-2 in terms of optimization of the coating material and process for making engineered interface between tungsten matrixes. The optimized interface material along with the process will be used to make compacts for the evaluation of various characterization tests as mentioned in Table 3.2.1. Samples will also be fabricated to characterize the developed Wf/W composite for its thermal fatigue test (sub size specimen) at IPR.

Fabrication and submission of samples (deliverables) to IPR and detailed project report on technology development will constitute Phase-2 of the MoU.

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3.0 DELIVERABLES:

3.1 Deliverables for Phase 1

- (a) Wf-W composite material samples Type-1 and Type 2 (2 nos. each) in coupon size.
- (b) Detailed report in hard copy as well as soft copy with all generated data to IPR.

3.2 Deliverables for Phase 2

(a) Wf/W composite samples with targeted properties with ± 10% standard deviation (as mentioned in Table 3.2.1) of size: width: 30 mm, breadth: 30 mm and thickness:
10 mm

- I. Potassium (K) doped tungsten fibre-tungsten composite (Type-1): 03 nos.
- II. Potassium (K) doped tungsten fiber-tungsten composite with engineered interface (coated fibers) (Type-2): 03 nos.

The targeted properties of the tungsten fiber-tungsten composite material is given in Table 3.2.1 below

Property	Target
Density	18 g/cc
Ultimate tensile strength (UTS)	450 MPa at 500 °C
Yield strength (YS)	400 MPa at 500 °C
Total elongation	15% at 500 °C
Flexural Strength	150 MPa at 500 °C
Fracture Toughness	Two-times or higher than that of the Pure Bulk Tungsten Material
Vickers hardness	400 HVN
Thermal conductivity	140 W/m.K at RT
Grain size	20 μm or finer

Table 3.2.1 Tungsten fiber-Tungsten composite properties;

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- 3.2.2 Thermal fatigue testing of Wf-W composite material will be carried out at IPR at 500 °C temperature for 1000 nos. of cycles for performance check and qualification of material.
 - (b) Detailed report in hard copy as well as soft copy with all relevant data to be submitted to IPR.

4.0 Time schedule of MoU:

The timeline for the MoU is tabulated below:

	Phase-1 (months)				Phase-2 (months)					
Activity	3	6	9	12	15	18	3	6	9	12
Procurement and characteri- zation of raw materials (in- cluding cathode materials for magnetron sputtering)	=									
Procurement of high density graphite dies	II	11								
Optimization of SPS parame- ters for fabricating Wf/W composite		8		8		8				
Optimization of coating pa- rameters			*	*		*				
Characterization of sintered product for its physical, me- chanical, microstructural and thermal properties			Æ	Ħ		Ħ	Æ	Æ		
Thermo-mechanical fatigue test at IPR								11.	<i>11.</i>	×
Analysis and comprehensive final report after phase-1 and phase-2					=					=

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5.0 GENERAL TERMS:

The general terms of this MOU are as below:

5.1 VALIDITY:

This MOU shall be valid for a period of 2.5 years (30 months) from the date of execution of this MoU or co-terminus with the project sanctioned by funding agencies.

5.2 FINANCIAL COMMITMENT:

IPR shall pay a total of Rs. 49.00 Lakh (Indian Rs. Forty-Nine Lakhs) with applicable taxes, if any - to ARCI in FOUR stage-wise instalments towards the developmental work of MoU as per following terms and conditions:

- First Part (initial payment) of Rs. 14.7 Lakh (30% of MoU cost) will be released by IPR on receipt of the Invoice and Indemnity Bond for equal amount after signing this MoU;
- II. Second Part of Rs. 14.7 Lakh (30% of MoU cost) will be released by IPR on receipt of Invoice for equal amount and on approval of the IPR authorities about successful completion of Phase 1 of MoU.
- III. Third Part of Rs. 9.8 Lakh (20% of MoU cost) will be released by IPR on successful completion of Phase-1 of MoU and on receipt of Invoice and Indemnity Bond for equal amount and on approval of the IPR authorities to initiate Phase 2 of the MoU.
- IV. Fourth and Final Part of Rs. 9.8 Lakh (20% of MoU cost) will be released by IPR on successful completion of Phase-2 of the MoU.
- All payments by IPR will be made only through either Cheque or Demand Draft or Wire Transfer.

The overall financial implications of the project are as follows:

S.No.	Description	Cost, Rs.
1	Raw materials and other consumables (includes tung- sten powder, K doped tungsten fibers, high density graphite dies and targets and chemicals for coating the tungsten fibers)	15,00,000
2	Machine utilization	15,00,000
3	Testing and characterization#	8,00,000

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4	Salaries*	6,00,000
5	Travel	50,000
6	Outsourcing (fabrication)	2,00,000
7	Contingency	2,50,000
8	Total	49,00,000/-

Rupees forty nine lakhs only + Taxes extra as per applicable rates

– Tests include the comprehensive performance and characterization of $W_{f}W$ composite, as mentioned in the proposal

* – Includes dedicated manpower

Justification of Costs:

- 1. Powders and other consumables: The high density graphite dies for both 100 kN and 1000 kN capacities are expensive and are usually imported from foreign sources. Given the reactivity of graphite dies for tungsten at high temperature and deflection of graphite at high pressures, the expected life of the graphite dies is minimal. Hence, substantial costs are involved towards the procurement of these graphite dies. Moreover, the K-doped tungsten wire is supplied a minimum of 5 km in length. The chemicals for engineering the interface of the tungsten fibre to get rare earth oxides are costly and also to be imported.
- 2. Machine utilization: The proposed project mandates the use of ARCI's SPS facility of both 100 kN and 1000 kN capacities for the consolidation of W_f/W composite. Again, given the extreme ranges of temperatures and pressures to be used and considering the mass of the graphite dies, the heating and cooling for a single cycle can take a full day, especially in 1000 kN SPS machine. The magnetron sputtering technique will be used for coating. Several trial runs have to be carried out on different oxide systems to arrive at the optimized coating parameters.
- Characterization costs: The costs involved towards the utilization of various characterization tools available at ARCI. This includes density / SEM / EBSD / XRD / micro hardness / thermal conductivity / tensile properties.
- 4. Outsourcing: High temperature property testing and preparation of samples for the testing. Removal of adherent WC from tungsten component after spark plasma sintering. The specimens for characterizing the thermal conductivity and tensile

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properties are to be EDM wire cut from the bulk of the developed composite and are costly.

5.2 INTELLECTUAL PROPERTY:

Intellectual property (both Background and foreground) as defined hereunder includes, but is not limited to inventions and ideas, whether patentable or not, copyrightable subject matter, data bases, data compilations and collections, technical data and information, process technology, computer programs, methodologies, plans, drawings etc. 'Background intellectual property' is the intellectual property held by either PARTY prior to this MoU in their respective fields. Such 'background intellectual property' will remain with the respective PARTY holding the intellectual property even after the expiry of this Agreement.

It is anticipated that research under this agreement may result in the creation of intellectual property (foreground intellectual property). Share of any new intellectual property so generated from the efforts under this MoU will be decided by both the PARTIES in writing at a later stage, as and when such new intellectual property is generated. No claim of IP shall be considered unless and until both the PARTIES agree on sharing of such new IP. Any publications arising from the activities under this MoU should be jointly owned by IPR & ARCI.

5.3 SECRECY & NON-DISCLOSURE:

Both the PARTIES hereby undertake to agree that the 'background intellectual property' of other PARTY shall be held confidential and will not be revealed to any third party without prior written permission from the other PARTY. The secrecy and non-disclosure also applies to both the parties for the information and intellectual property generated out of the scope of this MoU. Any such disclosure shall be made only with prior written consent from the either PARTY.

5.4 TERMINATION

Termination of this agreement shall be applicable on either of the following grounds:

- a) On non-realization of project activities;
- b) On completion of tenure of this agreement;
- c) By mutual consent and agreement between the PARTIES;

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d) By breach of the contractual obligations under this MoU.

5.5 AMENDMENT TO THIS MoU:

This MOU can be modified or revised only by mutual agreement in writing by IPR and ARCI. Any new additions to this MOU may be considered an annexure to this MOU.

5.6 TECHNICAL FOCAL POINTS:

The nodal officers acting as point of contact from ARCI for this MOU are:

- 1) Dr. Dibyendu Chakravarty, Scientist-E, CNM, ARCI-Principal Investigator (PI)
- 2) Dr. Krishna Valleti, Scientist-E, Center for Engineered Coatings, ARCI- (Co-PI)
- 3) Dr. R. Vijay, Scientist-G and Team Leader, CNM, ARCI-- (Co-PI)

Whereas the nodal officers acting as point of contact from IPR for this MOU are:

- Mr. Shailesh Kanpara, Scientific Officer-E, High Temperature Technologies Division (HTTD), IPR – Project Coordinator (PC);
- Mr. Alpesh K. Patel, Scientific Officer-E, High Temperature Technologies Division (HTTD), IPR –(Co-PC);
- 3) Dr. Samir Khirwadkar, Scientific Officer-H and Division Head, High Temperature Technologies Division (HTTD), IPR. - (Co-PC)

5.7 SETTLEMENT OF DISPUTES

In the events of any difference or dispute between the parties, arising out of implementation of such agreement, the same should be resolved as per Department of Public Enterprises OM No. 4(1)/2013-DPE(GM)/FTS-1835 dated 22.05.2018.

5.8 FORCE MAJEURE CLAUSE

Neither party shall be held responsible for non-fulfilment of their respective obligation under this MoU, due to the exigency of one or more of the force majeure events such as hurricanes, floods, earthquakes, tsunami and weather disturbances as "acts of God". And other events such as war, terrorism, civil disorder, labour strikes or disruptions, fire, disease or medical epidemic or out brakes, and any other events including emergencies or non-emergencies. The party affected by these shall be given notice in writing to the other

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party within one month of such occurrence and cessation. If the majeure conditions continue beyond six months, the parties shall then mutually decide about the future course of action.

IN WITNESS WHEREOF, the parties have caused this MoU to be executed in the English languages in duplicate by the proper officials as of the date hereof.

For and on behalf of IPR

For and on behalf of ARCI

1. 2 hatened. Director

Institute for Plasma Research

Date: 24/FEB/2021 Place: Gandhinagar

(sheilesh Kampiky) (Witness-1 of IPR):

Seal:

24 FEB 2021

(Witness-2 of IPR): (Dr. Samir Khirwedtan)

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International Advanced Research Centre for Powder Metallurgy and New Materials (ARCI)

Date: 02 /03 / 2021 Place: hyderabad Seal:

2/3/2021 2/3/2021 (W.L.VIJAM) (Witness-1 of ARCI):

(Witness-2 of ARCI):

(8 Ann) (8 Ann) 2/3/21

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