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Initial Temperature Testing of Advanced Manufactured Melt Wire Package

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ABSTRACT

As a part of the In-Pile Instrumentation Program, The Idaho National Laboratory (INL) has recently established in-house capabilities to fabricate and test new advanced manufactured sensors for measuring peak irradiation temperature inside a nuclear test reactor. Although nuclear test reactors can use standard methods of real-time temperature monitoring, such as thermocouples, the complexity of feedthroughs and attachment complications to collect real-time measurements greatly increases the cost of the experiment. Passive monitoring techniques may be utilized that exploit the melting point of well characterized material (standard melt wires), however, the limited space available for instrumentation during experiments is also a challenge. To accommodate this challenge, INL has expanded its temperature detection instrumentation capabilities paired with the advancement of in-house advanced manufacturing options to include printed melt wires for peak irradiation temperature measurements. The advanced manufactured melt wire package can determine peak temperatures in experiments that can fit the challenging space available during irradiation. Recent results and knowledge from this ongoing work have made it possible to develop unique temperature sensors that can accomplish user specific temperature ranges inside the harsh environments experienced during radiation testing. This report summarizes the initial temperature testing using printed melt wire lines with silver (Ag) nanoparticles that have a high melting point of ~960 °C. The printed lines were initially tested for their relative melting points using a non-standard method with a furnace under ambient conditions. Future work will include more controlled way of testing printed melt wires under inert gas with multiple temperature measurements followed by comparison of standard melt wire package performance for baseline characterization.

Key words:

Melt wire sensor; in-pile instrumentation, advanced manufacturing, printed sensor

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ACRONYMS

MTRs – Materials Test Reactors

ATR – Advanced Test Reactor

INL – Idaho National Laboratory

PIE – Post-Irradiation Examination

AM – Advanced Manufacturing

EP – Extrusion Printing

AJP – Aerosol Jet Printing

PJP – Plasma Jet Printing

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1. Introduction

The harsh environment within a nuclear reactor introduce unique challenges to materials such as fuels and structural components; as it requires that materials are capable of withstanding these extreme environments i.e. higher neutron fluence, higher operating temperature, and larger amounts of stress. To develop a better understanding for the effect of irradiation these materials must undergo rigorous testing in order to evaluate their performance under reactor conditions. The ability to investigate the neutron response of a material is facilitated by Materials Test Reactors (MTRs), such as the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL). While some experiments only study the material with Post-Irradiation Examination (PIE) to complete their research objectives, others require data collection inside the reactor during the irradiation testing. As a result, significant efforts towards research and development in sensor and instrumentation have provided tremendous advancements in the availability and reliability of instrumentation for irradiation tests in MTRs.

The Advanced Manufacturing (AM) portion of the In-Pile Instrumentation Program has been exploring novel technologies that would allow for the development of unique sensors that are not possible through conventional fabrication processes. The direct-write techniques of Extrusion Printing (EP), Aerosol Jet Printing (AJP) and Plasma Jet Printing (PJP), were identified as tools demonstrating significant promise for diversity of application which would allow for the production of a wide range of sensors that are not only miniature, but also robust. AJP and PJP are capable of creating complex 2D designs while utilizing an ever-growing list of materials, and these benefits provide significant potential for the development of innovative in-pile instrumentation. The inks used for AJP and PJP are made out of nanoparticles that can be used to form narrow (tens to hundreds of micrometers) and thin (typically hundreds of nanometers) lines in an arrangement desired by the researcher. In order for the print to be functional, it is sintered, which then creates wires and films that act as active or passive sensors.

Passive sensors, such as a melt wire package, send no signal and are visually examined post irradiation. These melt wire packages are capable of detecting peak temperature during a radiation test; where a material that has been well characterized for its composition and melting temperature is placed in a radiation test. Once the test is finished, the melt wire is visually examined to determine whether the melting temperature was achieved, thus providing the information on the maximum peak irradiation temperature achieved inside the reactor during irradiation testing. Standard melt wires are commonly used in test reactor experiments such as ATR [1-3]. However, some test designs have limited space due to the pre-designed capsules that are only a couple millimeters in diameter and contain multiple specimens at once; leaving no or very little space for instrumentation. The aforementioned direct-wire technologies provide significant benefits towards this challenge as they are an enabling technology for fabricating miniaturized melt wire packages. Finally, incorporating printing techniques for the development of advanced instrumentation and sensors significantly expands the design space while providing a method for rapid prototyping.

2. AM Printing Techniques for Melt Wires

Novel melt wire designs can address the challenge of limited spaces for experiments inserted in a nuclear reactor. Recently, a proof-of-concept melt wire "chip" was designed (**Figure 1**) at INL. This chip can provide an option for a miniaturized melt wire package that presents a prototype where the melt wire(s) are sheltered from a harsh reactor environment as they are sealed between two stainless steel disks. Briefly, a two-millimeter diameter stainless steel disk that was half a millimeter thick was milled to create a dimple or pocket. Melt wires were then printed within that pocket using AJP. A second steel disk was then laser welded to the first one in order to create a "melt wire chip". This melt wire chip can be easily placed within an experiment, regardless of the environment, as the wires are sealed within the steel disk "sandwich". One concern with the new design is the potential for these printed wires, that are created from nanoparticles dispersions, to poses a melting temperature that varies from the bulk counterparts. For the melt wires, it is critical that the melting temperature of the wire material be well characterized and measured accurately.

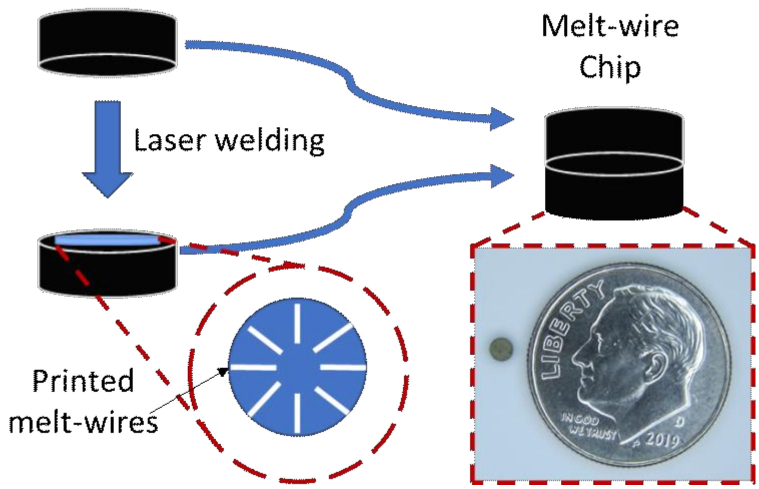

***Figure 1**- Schematic illustrating the melt wire printing and encapsulation.*

The printed melt wires require an understanding of the melting temperatures to demonstrate if there is a difference in melting behavior between standard and printed melt wires as well as providing a demonstration towards the ability to print multiple different materials capable of melting at different temperature ranges. For this, a low-melting point material ($Sn_{42}Bi_{57.6}Ag_{0.4}$) and a high-melting point material (Ag) were selected to print lines on a glass and alumina (Al_2O_3) substrates (**Figure 2(b) & (c)**), respectively. The direct-write technology chosen for this study was extrusion printing, and it was performed with a Voltera V-One PCB Printer by using a 225 µm nozzle (**Figure 2 (a)**). Only silver material was characterized for their relative melting temperatures to better understand if whether the particle size of the printed features play a role in the printed melt wire behavior. For each material an array design was printed consisting of three groups of three lines each with different lengths (1.0, 1.5 and 2.0 cm) that also varied in the number of print passes (1 pass, 2 passes, and 3 passes). The layout of the printed lines used for the initial temperature testing is shown in **Figure 2(b) & (c).** Finally, prior to initial temperature testing, the silver (Ag) lines were sintered at 200 ºC for 30 minutes, while the solder ($Sn_{42}Bi_{57.6}Ag_{0.4}$) was allowed to air dry overnight followed by a 50 ºC sintering for 30 minutes.

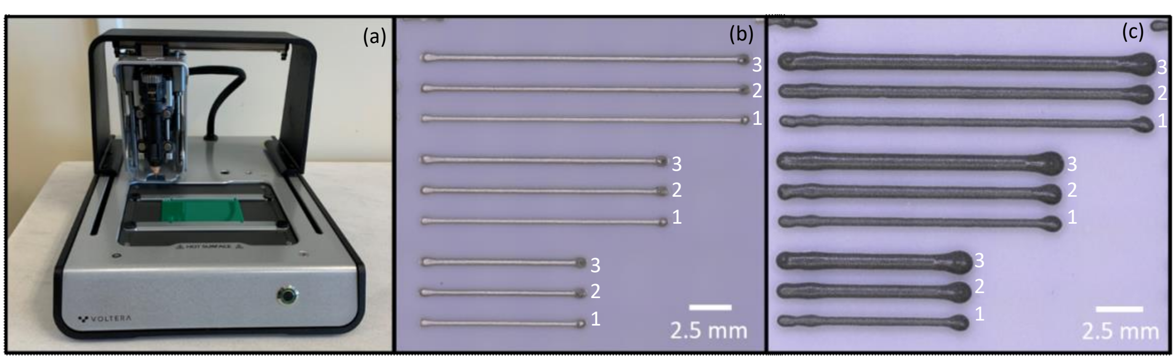

***Figure 2** – Printed Melt Wires: (a) Voltera V-One PCB printer used for printing both (b) Silver, and (c) Solder ($Sn_{42}Bi_{57.6}Ag_{0.4}$)*

3. Initial Testing Results

Initial temperature testing of printed melt wire array was conducted with a non-standard method by using a high temperature furnace under ambient conditions. This method used a standard furnace equipped with a digital camera and few thermocouples positioned at various points to record averaged melting temperature measurements (**Figure 3**).

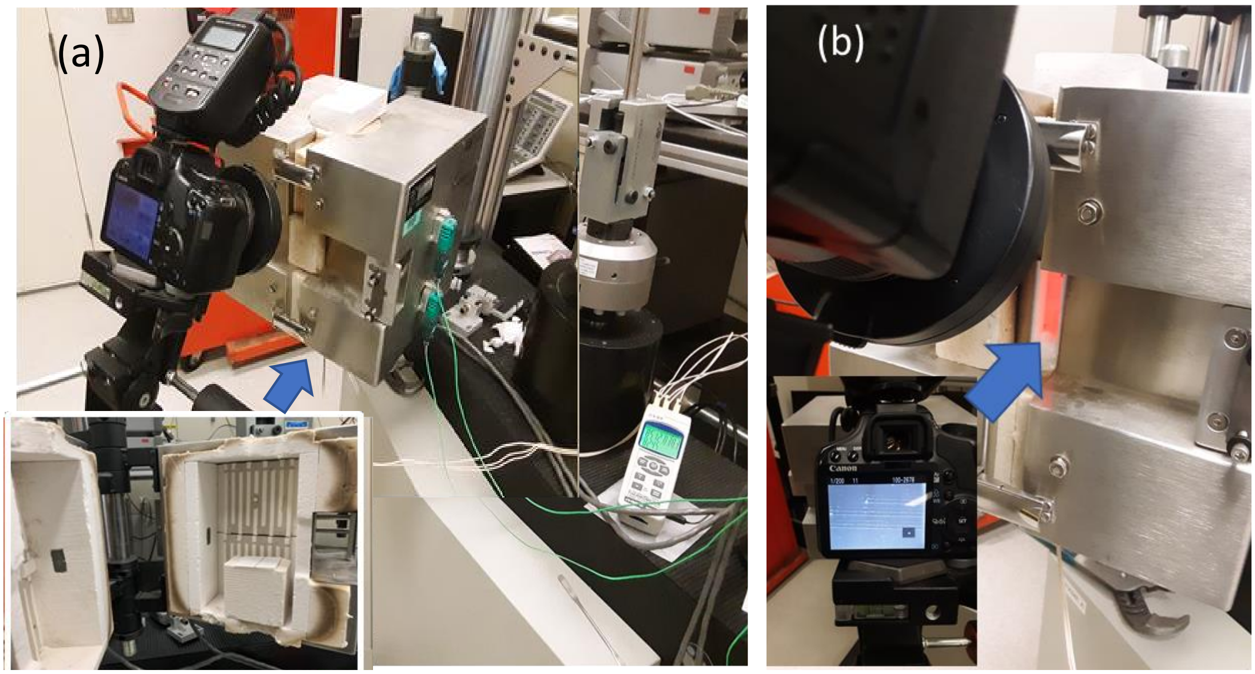

Figure 3 *– High Temperature Furnace Set-Up shown in (a) equipped with a digital camera and few thermocouples, and digital camera showing printed melt wire array inside the furnace as shown in (b)*

The silver melt wire array printed on an alumina substrate was placed inside the high temperature furnace (**Figure 4**). Since the thermocouples were not directly positioned onto the melt wire substrate a variation was noticed between both thermocouple measurements and only an average temperature was reported. Future work will seek to achieve a more precise way to measure temperature during melting under inert environment; hence, the reason why the printed solder ($Sn_{42}Bi_{57.6}Ag_{0.4}$) on glass with low-melting temperature was not used with this high-temperature furnace set-up.

***Figure 4**– Experimental Set-Up: (a) two thermocouples positioned under the alumna substrate, (b) printed silver melt wire array on alumina substrate, (c) magnified image of the substrate positioned inside the furnace, and (d) magnified image of printed silver melt wire lines on alumina substrate.*

The bulk metallic silver melts at around 960 ºC [4]; however, printed silver might melt at slightly different temperature from bulk due to the much smaller size of the silver nanoparticles [5]. The melting behavior of the printed silver melt wire array was observed at about 960 ºC (**Figure 5**). The printed silver lines broke and formed multiple droplets of agglomerated silver nanoparticles indicating the completion of melting at 960 ºC. The melted silver lines were analyzed post-melting under an optical microscope for their morphologies; the individual melted lines under microscope can be seen in **Figure 5(b) and (c)**. Since both, the bulk and nanoparticle printed silver, did not show any variation in melting temperatures further investigations and much more accurate way of measuring melting temperatures are necessary to fully understand the melting behavior of nanoparticle printed material vs. bulk material.

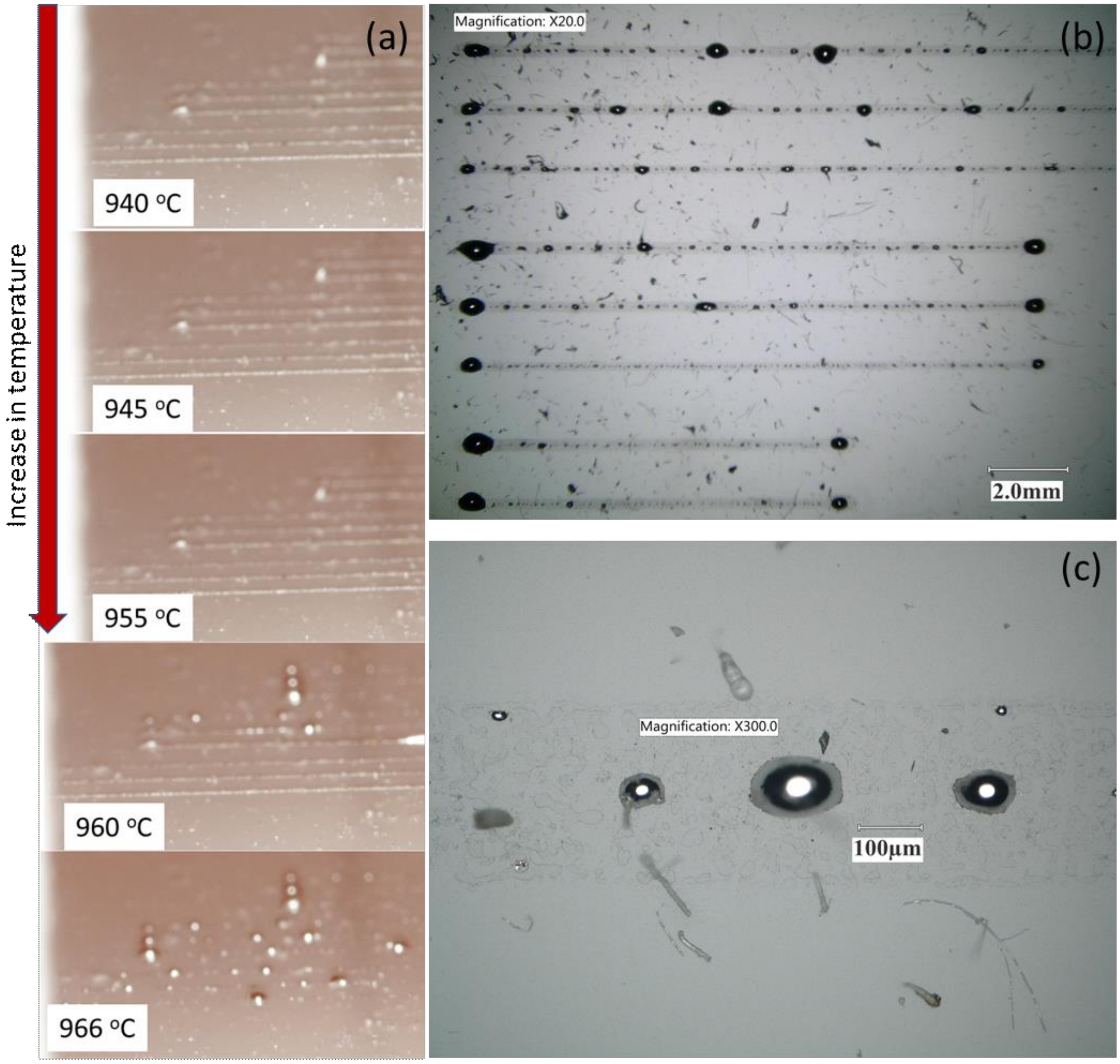

Figure 5 *– Melting process of printed silver lines with digital camera shown in (a), and under digital microscope post-melting in (b) & (c).*

4. Summary and Conclusions

INL has enhanced its melt wire capabilities with the introduction of advanced manufacturing techniques. It has been demonstrated that utilizing these techniques enable the support of irradiation testing within ATR and other test reactors having challenging space limitations. With the use of direct-write techniques, a peak temperature sensor has been fabricated that is two millimeters in diameter and one millimeter in thickness. A noteworthy benefit of these chips associated with miniaturization of the melt wire is the ability for one chip to contain multiple melt wires that have different compositions and, hence melting temperatures. Furthermore, encapsulation of the melt wires provides protection from potentially corrosive and harsh environments. Initial testing has been performed outside of the test reactor to validate not only the manufacturing procedure, but also the ability to visualize the melting behavior for the printed sensors. This report summarized the initial temperature testing using fabricated melt wires for silver (Ag) with high-melting temperature of 960 °C, that were initially tested for their relative melting points using non-standard method of a furnace under ambient conditions. Future work will incorporate a more controlled method of testing printed melt wires, which would include testing under inert gas with multiple precise temperature measurements. Finally, a comparison will be made between the response of a printed melt wire package and a classical melt wire package for baseline characterization purposes.

5. Acknowledgement

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