Microwave ignition for nanostructured reactive composites

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Abstract—

The need for heating at nanoscale has pushed researchers in the study of reactive, nanostructured composites known as nanoheaters. Major topics of interest are the best conditions for consolidation, composition, and ignition of these innovative heat sources. This work presents a new method of ignition for nanoheaters, known as non contact microwave ignition, distinguishing itself from previously developed direct heat application methods. Al-Ni nanoheaters were fabricated through ultrasonic powder consolidation (UPC) with embedded aluminum and copper wires. The conductive properties of the embedded wires, acting as susceptors when exposed to electromagnetic radiation in the microwave range, were found to induce enough heat to Al-Ni nanoheaters to facilitate ignition. This nullifies the requirement of direct heat application to the fabricated nanoheaters to produce ignition. In addition to testing in gaseous environment, this new method of ignition for nanostructured, reactive composites was also tested in vacuum, verifying its effectiveness in a non-gaseous environment.

I. INTRODUCTION

Nanoheaters are nanostructured reactive composites that allow localized and controlled heat generation when ignited. The advantages of providing controlled heating to a designated volume [1] through the use of this new form of reactive materials, have brought major advancements to a vast number of fields and processes such as microscale joining, microelectronics soldering [2], reshaping of parts for environmental degradation and ablation of biomaterials of cancer cell walls [3].

Current ignition methods of nanoheaters include continuous heating through a heater plate [2], direct heating through laser pulses or plasma torch [3] and direct current passing through the nanoheater [4]. The common aspect among the above ignition methods is the need of direct supply of applied heat to the nanoheater for ignition to occur, which restricts a lack of application to manufacturing processes where direct contact between the nanoheater and the heat source is not feasible. Today's industrial applications of nanoheaters highlight the need for a non-contact, indirect heating method for the ignition of these nanosized reactive materials.

In this paper, a newly developed non-contact microwave ignition method is presented. Leveraging the conductive properties of metals, the fabricated nanoheater with an embedded conductive wire will spark when exposed to electromagnetic radiation. The mechanism behind the conductive wires acting as susceptors for the nanoheater was investigated by subjecting aluminum and copper wires to electromagnetic radiation and observing their breakdown and sparking patterns. The consolidated nanoheaters with embedded aluminum and copper wires were tested in air and vacuum environments. By eliminating any form of direct application of heat to ignite nanoheaters, the non-contact microwave methodology enables the application of nanoheaters to manufacturing processes where controlled temperature and complex geometries are not to be seen as limitations [4].

II. EXPERIMENTAL PROCEDURE

The fabrication of the Al-Ni nanoheaters with conductive embedded wires was divided into three steps.

A. Dimensioning of wires

Aluminum and copper wires to be embedded in the nanoheaters were cut to size to investigate their properties as susceptors when exposed to microwave radiation. Two aluminum and copper spools with a wire diameter of 0.405 mm were used in the experiment. The wire spools were purchased from Malin Co. and Arcor Inc., respectively. A Mitutoyo Electronic Caliper was used to accurately measure the sections of conductive wire. A standard box cutter was used to cut the wires. Table I below shows the lengths of wires used in the experiments:

TABLE I
LENGHTS OF ALUMINUM AND COPPER WIRE SECTIONS

	Length #1	**Length #2**	**Length #3**	**Length #4**
Copper	20 mm	25 mm	30 mm	35 mm
Aluminum	24 mm	30 mm	35 mm	-

Due to the cross section of the wires and the tools used to cut them to size, the radius of the tip of each cut section could not be controlled. Overall, the goal was to cut the wires such that the tip radius resulted to be less than or equal to half of the wire diameter.

B. Mixing process

Aluminum and nickel nano-flakes with sub-micron thickness (100-300 nm) were supplied by Fukuda Metal Foil & Powder Co., Ltd. The aluminum and nickel nano-flakes were mixed with a 1:1 molar ratio. The mixing process for the consolidation of the nanoheaters consisted of three steps: dry mixing in air, mixing in ethanol and sonication in ethanol [1]. Table II below summarizes the corresponding times for each step of mixing:

TABLE II
DURATIONS FOR THE THREE STEPS OF NANO-FLAKE MIXING

Dry mixing in Air	Mixing in Ethanol	Sonication in Ethanol
60 min	60 min	120 min

A standard rotary powder mixer and a FS20D Sonicator by Fisher Scientific were used for the mixing process. The blended powders, once sonication was complete, were then left to dry until total evaporation of ethanol and dry mixed for 20 minutes before being used in the consolidation of the nanoheaters. The above three steps of mixing were followed to achieve better deagglomeration and reduction of aluminum and nickel nano-flakes clusters [1].

C. *Consolidation of nanoheaters*

The metastable state of nanoheaters requires a fabrication methodology that does not employ the use of high temperatures [1]. Ultrasonic powder consolidation (UPC) was employed in this experiment, being a new manufacturing technique that allows powders to metallurgically consolidate when subjected for a few seconds to ultrasonic vibration and low or moderate temperatures. The ultrasonic welder STAPLA Condor® used is a 3 kW setup with a fixed frequency of 20 kHz and an amplitude of vibration at the sonatrode of 9 μm [2]. Additionally, the UPC setup employed a heater plate to perform the consolidation of the specimens at elevated temperatures.

A set of a die and a punch was used to consolidate the nanoheater, Fig. 1. The die had dimensions of 12.4 mm x 12.4 mm x 4 mm with a through hole 6.30 mm in diameter, and the punch was a disk of matching diameter of 6.3 mm. Both the die and punch were made of mild steel.

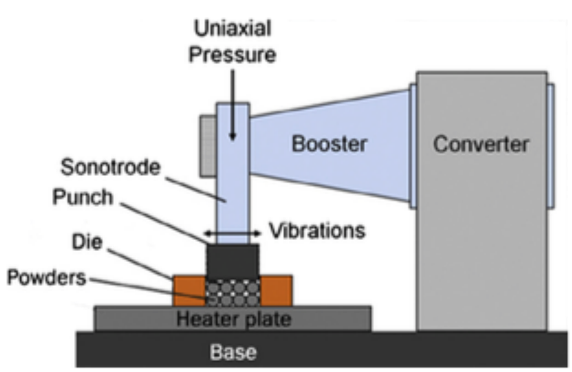


Fig. 1. Schematic of the STAPLA Condot UPC setup

The die with an aluminum or copper wire was put in place on the UPC setup and the consolidation temperature was set to 573 K. As the heaters reached the final temperature, approximately 0.1-0.2 grams of previously mixed aluminum and nickel nano-flake mixture was put in the die and a uniaxial pressure of 100 MPa was applied to it through the punch. Once the final temperature of 573 K was reached, 1 second of in-plane vibration was applied, Fig. 2. The consolidated Al-Ni nanoheater with embedded conductive wire, Fig. 3, was rapidly taken out of the punch and the heater turned off. This procedure was used for the consolidation of all nanoheaters used in the experiments of this paper.

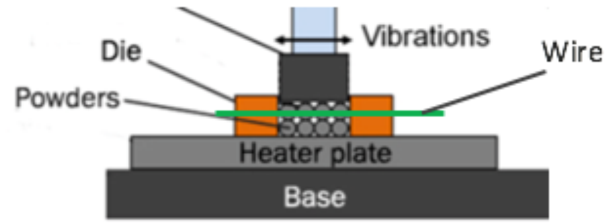


Fig. 2. Schematic of the punch and die for the consolidation of Al-Ni nanoheaters.

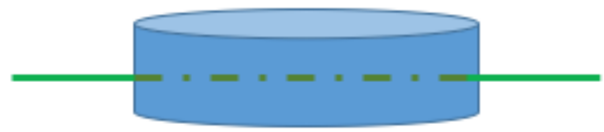


Fig. 3. Schematic of the punch and die for the consolidation of Al-Ni nanoheaters.

D. *Encapsulation of nanoheaters*

To observe the ignition of the consolidated Al-Ni nanoheaters with embedded conductive wires in a vacuum environment, the nanoheaters were encapsulated in such an environment using Pyrex tubes, Fig. 4. A single nanoheater was inserted in the Pyrex tube closed at one end and attached to a Platinum JB pump on the open end. The pump evacuated the Pyrex tube at a rate of 1.5×10^{-3} m^3/s, to a pressure of 4×10^{-4} mmHg [1] for 20 minutes. Once evacuation was complete, the open end of the Pyrex tube attached to the vacuum pump was sealed by heating with a propane torch.

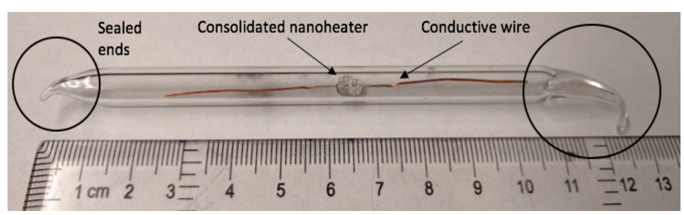


Fig. 4. The encapsulated nanoheater in a vacuum environment.

III. RESULTS AND DISCUSSION

A. Length of wire and time of first spark correlation

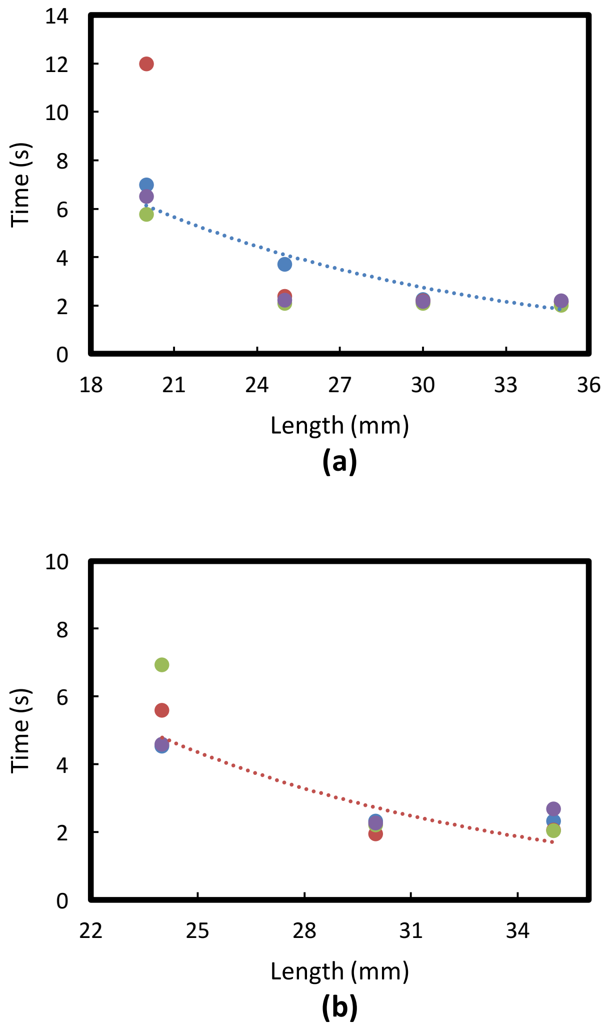


Fig. 5. Time of first spark leading to electrical breakdown of wire when exposed to microwave radiation (a) copper wire (b) aluminum wire.

Fig. 5 shows the correlation between the time of first spark and length for both copper and aluminum wires subjected to microwaves in air. In this experiment, the time of first spark was defined as the instance at which the first spark is seen on the conductive wires exposed to microwave radiation, leading to their complete melting and breakdown. The time of first spark was found to decrease with increasing wires length. It was noticed that for copper and aluminum wires, below l <20 mm and l <24 mm respectively, no sparks occurred on the wires and therefore no breakdown of the wire, when exposed to microwave radiation. All wires were exposed to the electromagnetic radiation generated by a 1000-Watt commercial microwave with a 2.45 GHz chamber. Each piece of wire was placed on ceramic substrate and then placed in the microwave chamber.

A plausible explanation for the sparks observed on the wires, leading then to their melting, can be given in terms of the corona discharge phenomenon. A corona discharge is a release of static charge caused by excessive ionization of a gas surrounding a sharp electrode. The instance of excessive ionization of a gas physically means that current is conducted through an ionized gas. In the experiment, as the microwave irradiation increases the potential of the wire, the molecules of the gas are also ionized, leading then to a release of the accumulated static charge. The release of static charge is the evidence of a corona discharge, seen by naked eye in the form of luminous and audible sparks.

The value of ionization at which the gas starts conducting current is defined as "electric breakdown potential" of the gas [1]. The electric breakdown potential of air has a value of is $3\text{x}10^6$ V/m. The equation for the electric breakdown potential of a gas is shown below:

$$E = \frac{Q}{4\pi\varepsilon_0 r} \tag{1}$$

where Q is the value of free charge to be calculated as a function of wire volume, ε_0 is the permittivity of free space constant with value of $8.852\text{x}10^{-12}$ F/m and r is the radius at the two ends of the wire. When the electric breakdown of air occurs, the conductive wires producing sparks melt and break up into smaller sections that are below the minimum sparking length. Fig. 6 below shows copper and aluminum wires after complete breakdown:

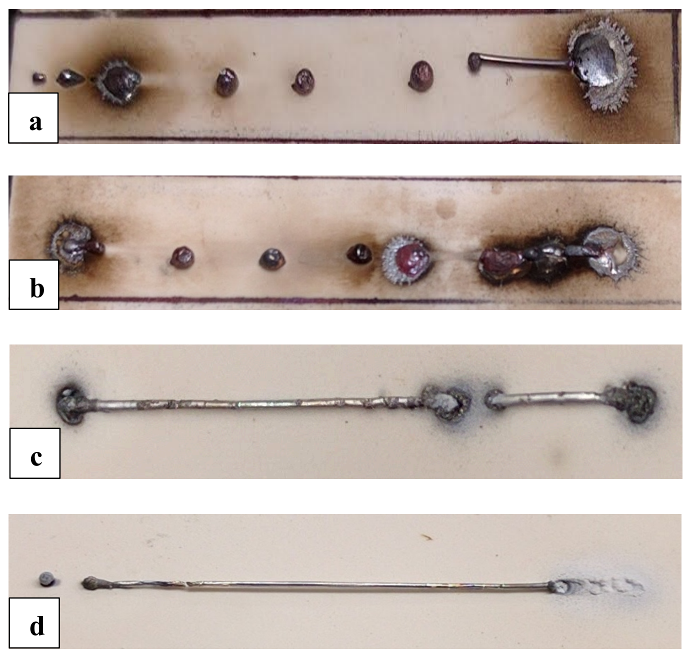


Fig. 6. Wires after being exposed to microwave radiation until complete electrical breakdown (a) 30 mm copper (b) 35 mm copper (c) 30 mm aluminum (d) 35 mm aluminum.

The melting and separation observed in the mechanical breakdown patterns of the conductive wires indicates that heat is generated in the wires. The evidence presented above, supports the hypothesis that the conductive wires, acting as susceptors, absorb the electromagnetic energy. As more electromagnetic energy is accumulated, the potential of the wire increases, causing a current flow. Any form of current flow through a conductor having a resistance, will cause heat generations.

This form of heat generation, presumed to be the driving force of this non-contact ignition methodology, is a form of Joule Heating. The equation of Joule heating is shown below:

$$H = \frac{1}{j} i^2 R t \tag{2}$$

Where j is a constant, known as Joule's mechanical equivalent of heat with a value of 4.1860 J•cal^{-1}, i is the current flowing through the conductive wire, R is the resistance of the wire and t is the time of current flow.

In order to have current flow along the wire, a potential difference must exist between the two wire ends. Therefore, it is presumed that as the sparks appear on the wires, a high voltage difference is created between the two tips, subsequently causing current flow.

We can calculate the theoretical voltage in the wire assuming that current flow starts at the instant of electrical breakdown of the ionizing gas:

$$V = E \cdot r \tag{3}$$

where E is the electrical breakdown potential of the gas surrounding the conductive wire and r is the radius of cut at the two tips of the wire. For the conductive wires in Air and with a radius of cut of 0.2025 mm we calculate a voltage of:

$$V = \left(3 \cdot 10^6 \frac{V}{m}\right) \cdot (0.2025 \cdot 10^{-3}) = 607\ V \tag{4}$$

Further on, the theoretical values of resistance for the smallest wires exposed to microwave radiation were calculated as:

$$R_{20mm\ Cu\ Wire} = 0.00260\ \Omega \tag{5}$$

$$R_{24mm\ Al\ Wire} = 0.00525\ \Omega \tag{6}$$

Finally, the theoretical amount of energy (in Joule) generated in the conductive wires when exposed to microwave radiation and therefore leading to the ignition of the nanoheaters, can be calculated as shown below:

$$J_{Copper\ wire} = \frac{V^2}{R} = \frac{(607)^2}{0.00260} = 1.417 \cdot 10^8 \quad \text{J/s} \tag{7}$$

$$J_{Aluminum\ wire} = \frac{V^2}{R} = \frac{(607)^2}{0.00525} = 7.018 \cdot 10^7 \quad \text{J/s} \tag{8}$$

Based on previous experiments and related calculation conducted by Gheybi [1], the minimum amount of energy required for the ignition of bi-metallic, 2Al-Fe_2O_3-3(Al-Ni) nanoheaters was calculated. The same non-contact ignition methodology used in Gheybi's experiments demonstrated that the embedded copper and aluminum conductive wires, produced enough energy for the ignition of the nanoheaters. The section below explores the efficiency of this nanoheaters ignition methodology, proving its effectiveness for the ignition of Al-Ni nanoheaters both in gaseous and non-gaseous environments.

B. Nanoheaters ignition in air

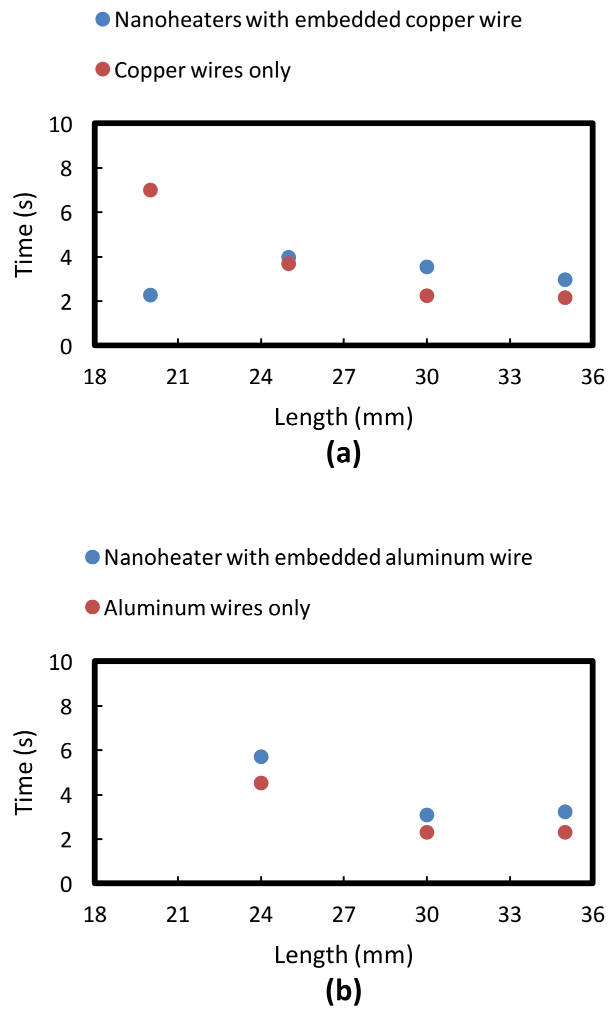
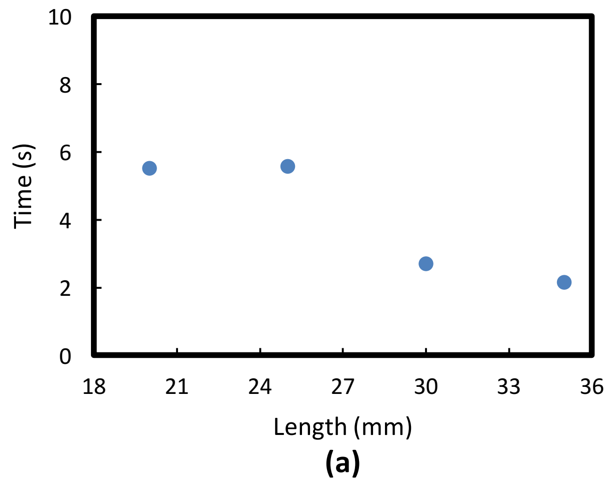


Fig. 7. Nanoheaters ignition time with embedded wire in Air (a) copper wire (b) aluminum wire.

Fig. 7 shows the effectiveness of the non-contact nanoheater ignition methodology. The fabricated nanoheaters were exposed to microwave radiation in a gaseous environment, air, all igniting on the first try. From the graphs, it is noticeable that the times of ignition of the nanoheaters, correspond to the times of first spark on the conductive wires tested in section A above. The results support and prove the hypothesis that there is current flow in the wires the moment sparks appear, resulting in enough heat generation to produce the ignition of the nanoheater.

C. Nanoheaters ignition in vacuum



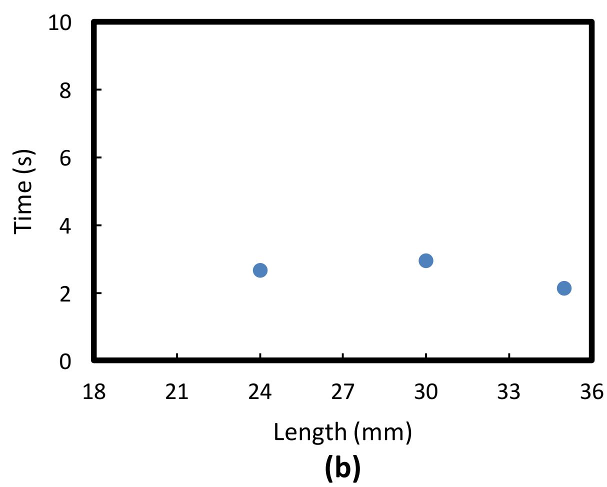


Fig. 8. Ignition times of Al-Ni nanoheaters with embedded conductive wire when exposed to microwave radiation in vacuum (a) copper wires (b) aluminum wires.

To further prove the effectiveness of the non-contact microwave ignition method in non-gaseous medium, the fabricated Al-Ni nanoheaters were ignited in vacuum. Fig. 8 describes the ignition times of Al-Ni nanoheaters with embedded wires of different lengths when exposed to microwave radiation in a vacuum environment. In vacuum, the ignition characteristics and mechanism of the nanoheaters remain unchanged. Current flowing through the conductive embedded wires, which generates enough heat for the ignition of the nanoheaters, is still the supported hypothesis in the tested non-gaseous environment. In Gheybi's work [1], the ignition of nanoheaters in vacuum is hypothesized to occur at Schwinger's Limit. In Quantum Electrodynamics (QED), the value of Schwinger limit is of $1.3x10^{18}$ V/m, representing the point at which an electromagnetic field becomes non linear [5]. Under such conditions, the dielectric of a material responds non linearly to an applied electric field.

In the above condition, the lack of a gas surrounding the conductive wires, automatically excludes the possibility of ionization and electrical breakdown of air as the leading cause for sparks to occur on the wire surface. The major difference in the tested non-gaseous environment is the way electrical breakdown is achieved. The early stages of vacuum breakdown can be justified by the cold electron emission phenomenon. This phenomenon causes electron emission by exposing the impurities and imperfections on the surface of the wires to a high electric field. Electron collisions, the essence of current flow inside the wires, generate heat due to Joule heating, leading to the melting of impurities and release of hot electrons. This chain of electron emissions is the base of the electron avalanche concept, also known as Townsend discharge [6], [7]. The constant heat generation finally leads to the breakdown of the wire and ignition of the nanoheaters.

IV. CONCLUSION

In conclusion, reactive Al-Ni composites were ultrasonically consolidated from Al and Ni nano-flakes with embedded Aluminum and Copper conductive wire. The consolidation of the nanoheaters occurred at a temperature of 573 K under 100 MPa of pressure. It was observed that the fabricated nanoheaters ignited within seconds without the need of direct heat application when exposed to microwave irradiation of a conventional 1000 Watt, 2.45 GHz microwave oven.

The optimal conditions for the ignition of nanoheaters with embedded conductive wires was observed in vacuum. The Al-Ni nanoheater with 35 mm embedded copper conductive wire ignited in 2.16 seconds, when exposed to microwave irradiation. Additionally, the fastest ignition in vacuum was produced by nanoheaters with embedded aluminum wires.

From the above experiments, it is hypothesized that in a gaseous environment, as dielectric breakdown of the gas is achieved and that the release of static charge, by means of corona discharge phenomenon, induces current flow in the conductive wires, generating enough heat for the ignition of the nanoheaters. In vacuum, it is hypothesized that due to Townsend electron avalanche, the chain of electron emission caused by the exposure of the impurities in the material to a high electric field, induces current flow in the conductive wires, resulting in enough Joule heat generation for the ignition of the nanoheaters.

Further work is required for a deeper understanding of ignition characteristics of the nanoheaters with conductive embedded wires when exposed to microwave irradiation. A broader understanding of the nanoheater's ignition mechanism with conductive embedded wires could enable us to fabricate more compact, efficient and advanced nanoheaters. Possible applications of more advanced nanoheaters and ignition methodologies could have lasting impacts on electronics soldering, ablation of cancer cells and similar fields where localized heat generation by means of indirect heat application to the heat source is required.

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