



# U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND – ARMAMENTS CENTER

## A New Class of Pyrophoric Foam Materials: Synthesis and Characterization

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# INTRODUCTION



## BACKGROUND:

- Finely powdered iron metal particles are pyrophoric, which is largely a function of its particle sizes.

## APPLICATIONS:

- Cathode materials for fuel cells
- Active agent in chemical sensing device
- Catalysts for ammonia synthesis, liquid-phase hydrogenation, and groundwater remediation, etc.
- Once passivated, used as pigments for magnetic tapes and in medical practice for its bacteriostatic properties.

## THE CHALLENGES:

- Aggregation and sintering of nanosized Fe<sup>0</sup> particles, resulting in lost reactivity

## PRIOR EFFORTS:

- Efforts to segregate and stabilize nanosized iron particles on substrates with large surface areas,
- Such as metallic foils, non-combustible fiber meshes, activated carbons, zeolites, and, recently, carbon nanotubes, etc.

## THE ISSUES UNRESOLVED:

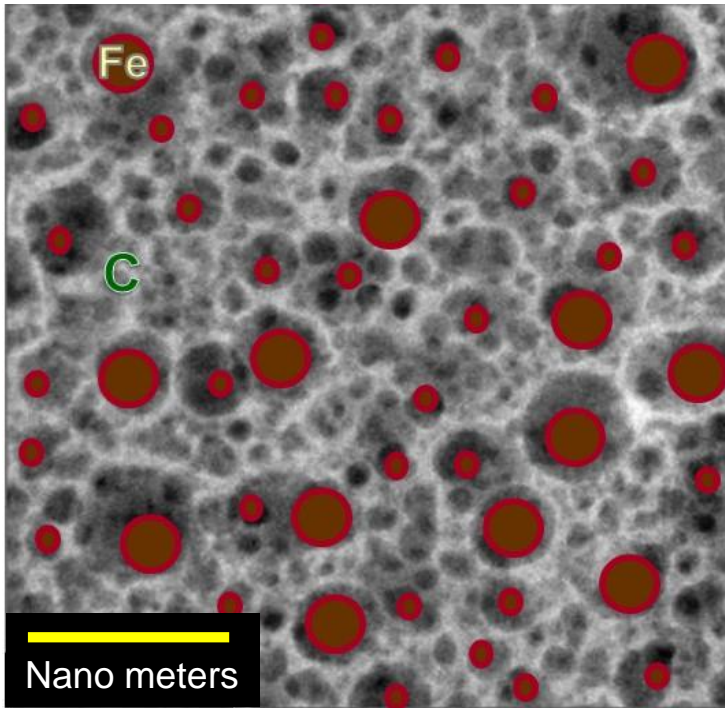
- No nano-scale confinement
- Limited loading of active iron ingredients
- Dusty composite products
- Delicate and greatly compromised thermal activation processes



## OBJECTIVE



### Make highly pyrophoric foam materials



**FIGURE 1.** Illustration of Nanosized Fe<sup>0</sup> Particles in Nanoporous Carbon Matrix

### Make highly pyrophoric foam materials

#### PROCESSES:

- A simple one-pot synthesis route via
- A combination of two known processes for pyrophoric Fe<sup>0</sup> particles and carbon foams.
- Therefore, the synergy between these two reactions (proceed concurrently) is exploited.

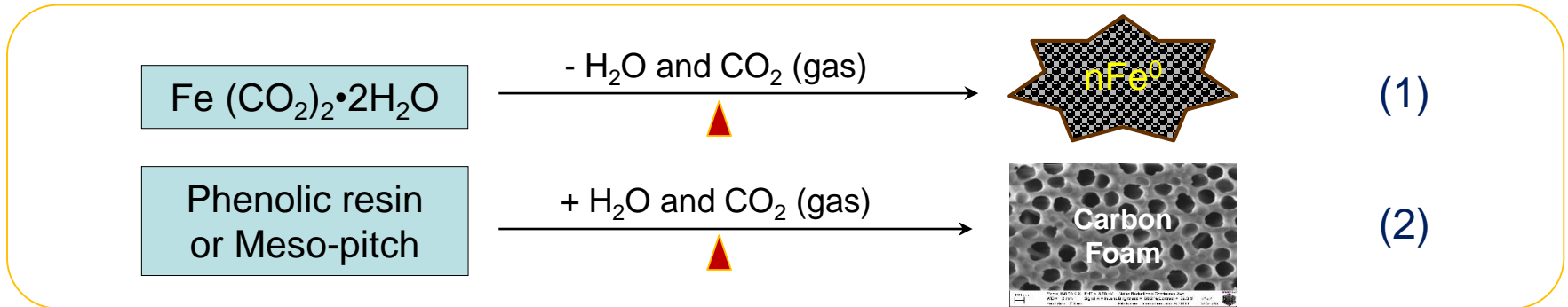
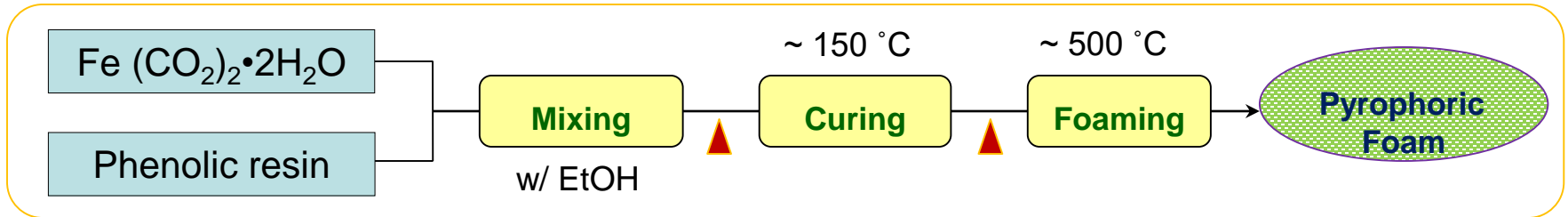
#### PRODUCTS:

- Carbon foam is tailor-made through the formation of Fe<sup>0</sup> particles in its matrix, while itself is also flammable and contributes to the overall pyrophoricity of the material.

*Luan Z.; Mills, K.C.; Morris, L.A.; Haines, C. D. U.S. Patent 10 059 637, 2018.*



# EXPERIMENTAL



## TYPICAL EXAMPLE:

### Raw materials:

- Iron oxalate dihydrate
- Phenolic resin (PG-5520)
- EtOH (200 proof)

### Process parameters:

- Mixing, casting and drying in air
- Curing at 160 °C
- Foaming and activation at 500 °C in Ar/H<sub>2</sub>

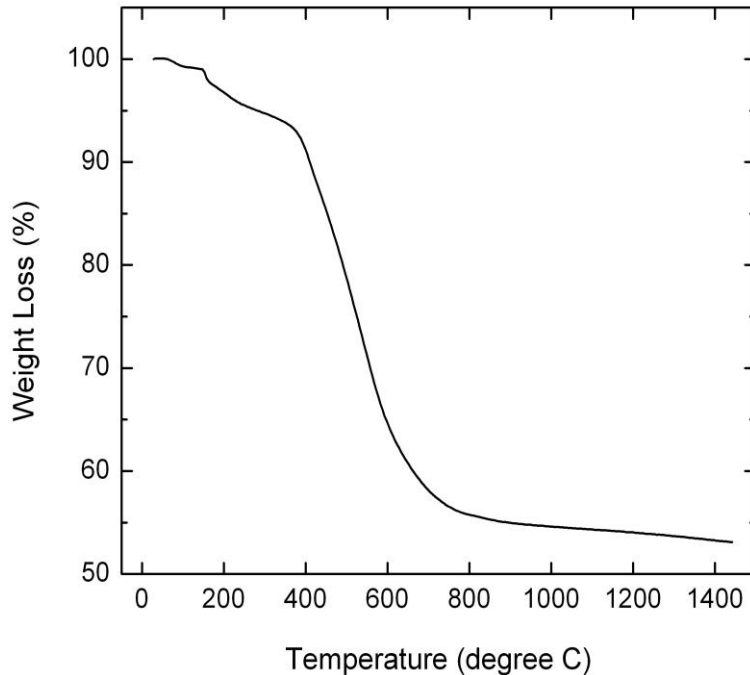


## RESULTS - ACTIVATION

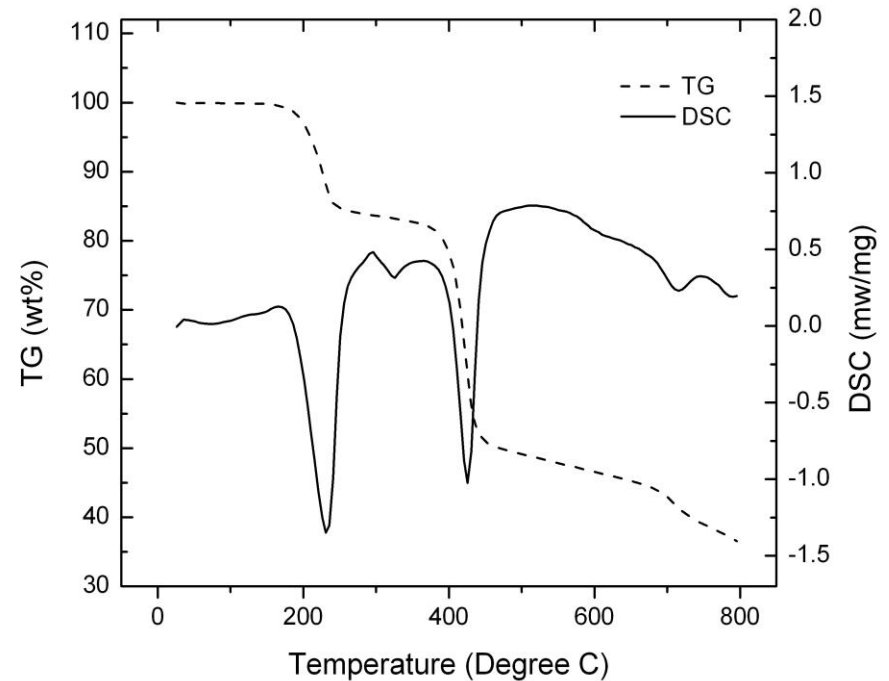


### Materials produced at 500 °C in Argon

#### *Phenolic resin*



#### *Foam materials*



**Figure 2.** Thermogravimetric (TG) and differential scanning calorimetry (DSC) measurement of the activation of pure phenolic resin and foam materials in Argon.

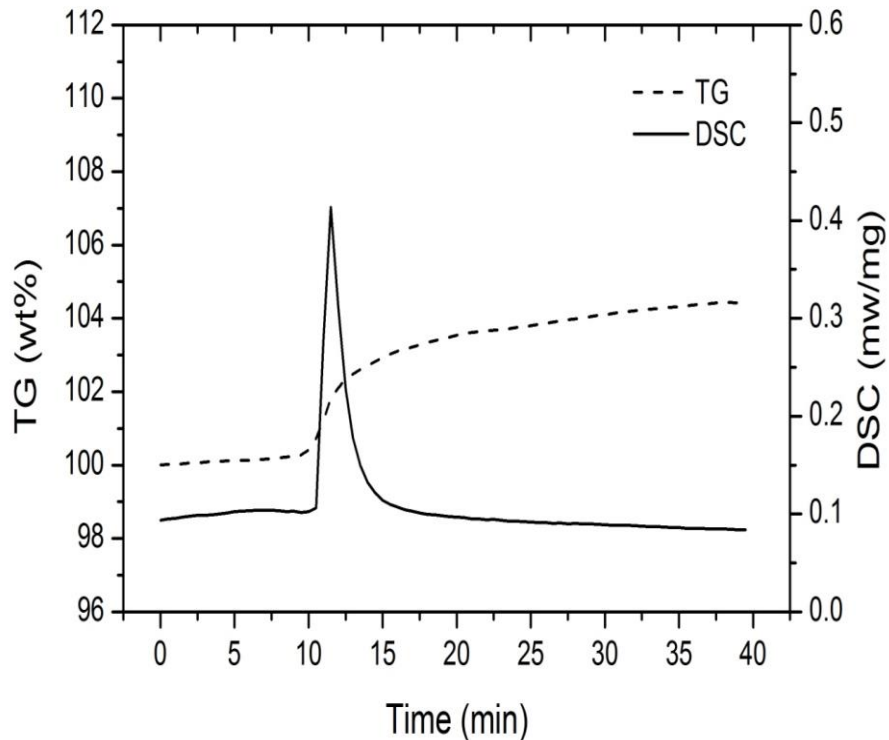


## RESULTS - PYROPHORICITY

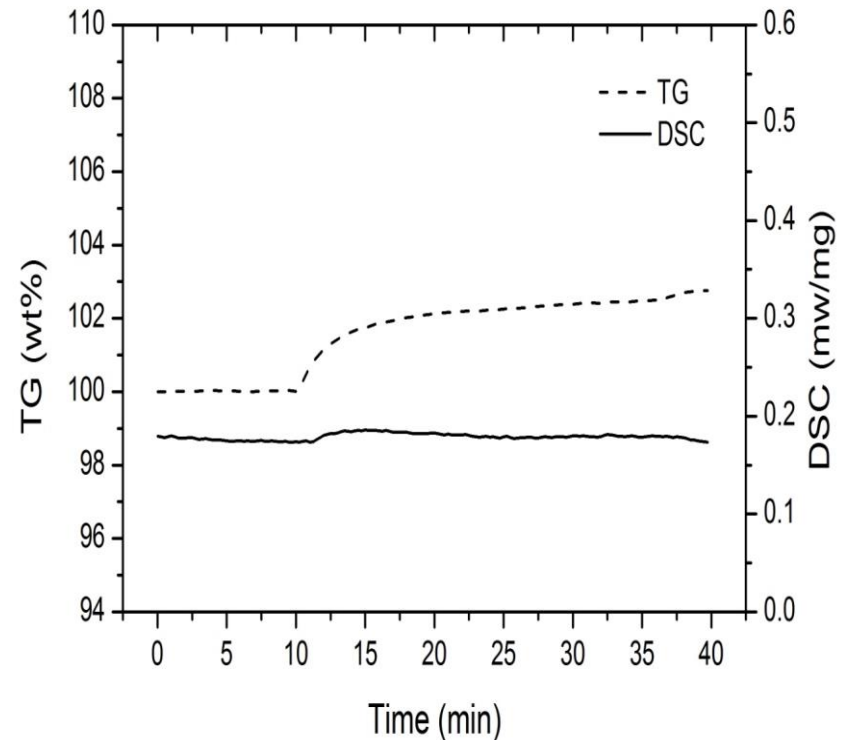


### Materials produced at 500 °C in Argon

#### *Foam materials*



#### *Powder materials*



**Figure 3.** Thermogravimetric (TG) and differential scanning calorimetry (DSC) measurement of materials produced upon exposure to air flow at ambient condition.

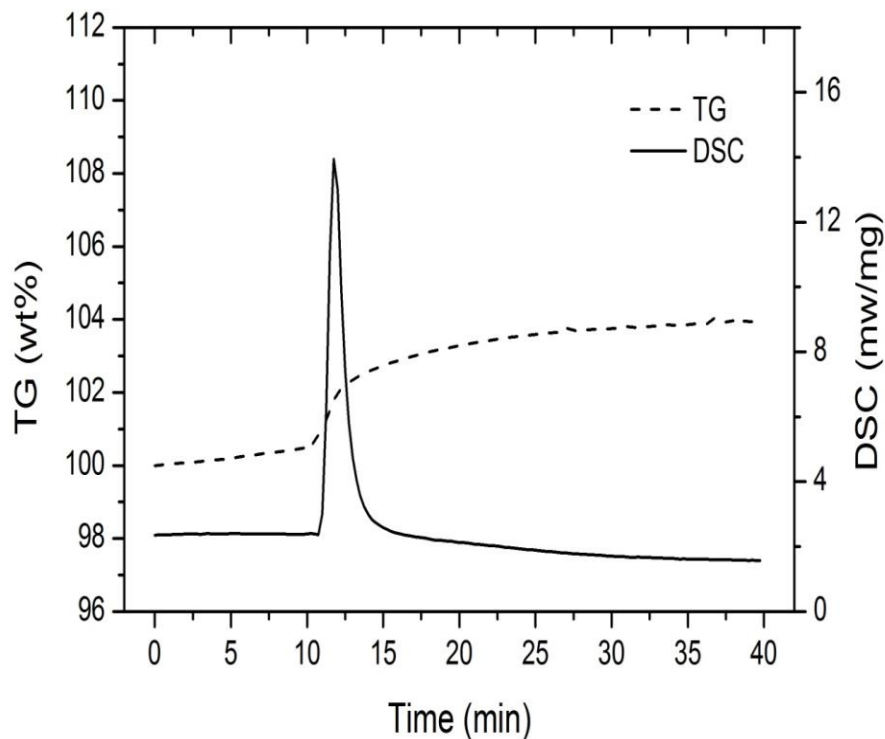


## RESULTS - PYROPHORICITY

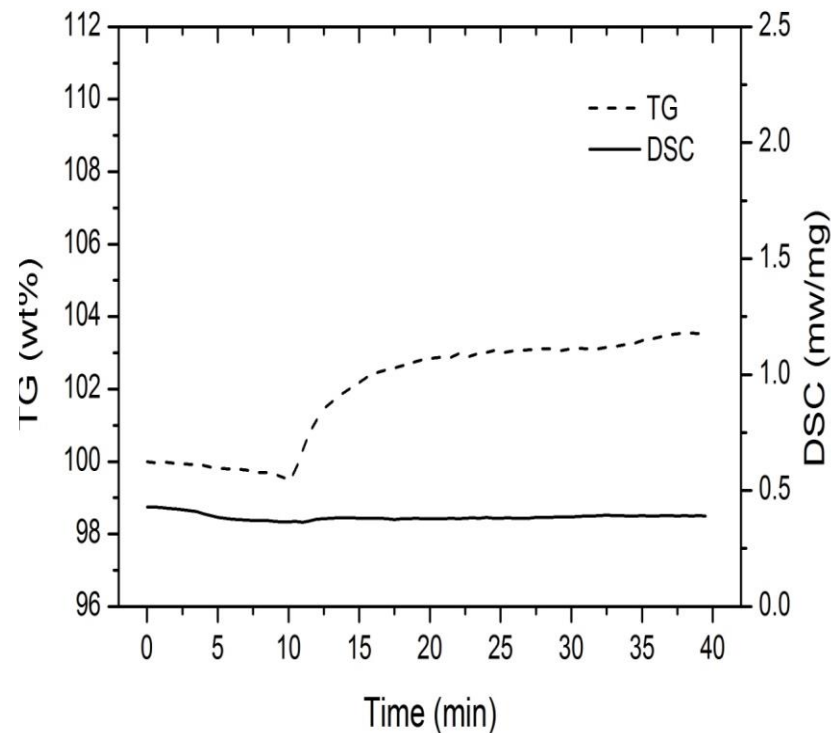


Materials produced at 500 °C in 5% hydrogen in Argon

*Foam materials*



*Powder materials*



**Figure 4.** Thermogravimetric (TG) and differential scanning calorimetry (DSC) measurement of materials produced upon exposure to air flow at ambient condition.



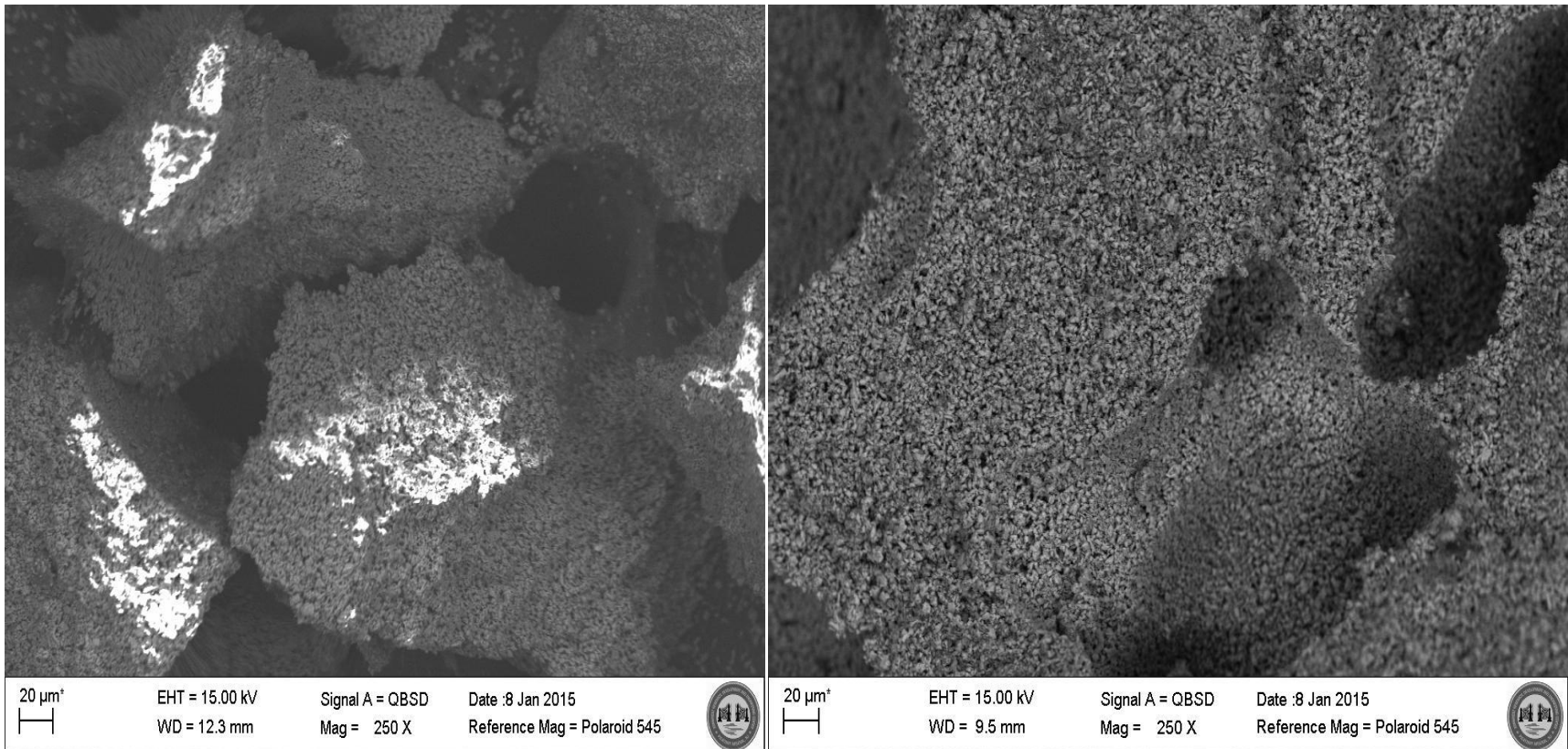
## RESULTS



## Foam materials produced at 500 °C in 5% hydrogen in Argon

*Cured*

*Activated*



**Figure 5.** Scanning electron microscopic (SEM) images of the foam materials cured at 160 °C (left) followed with activation at 500 °C in Argon (right).



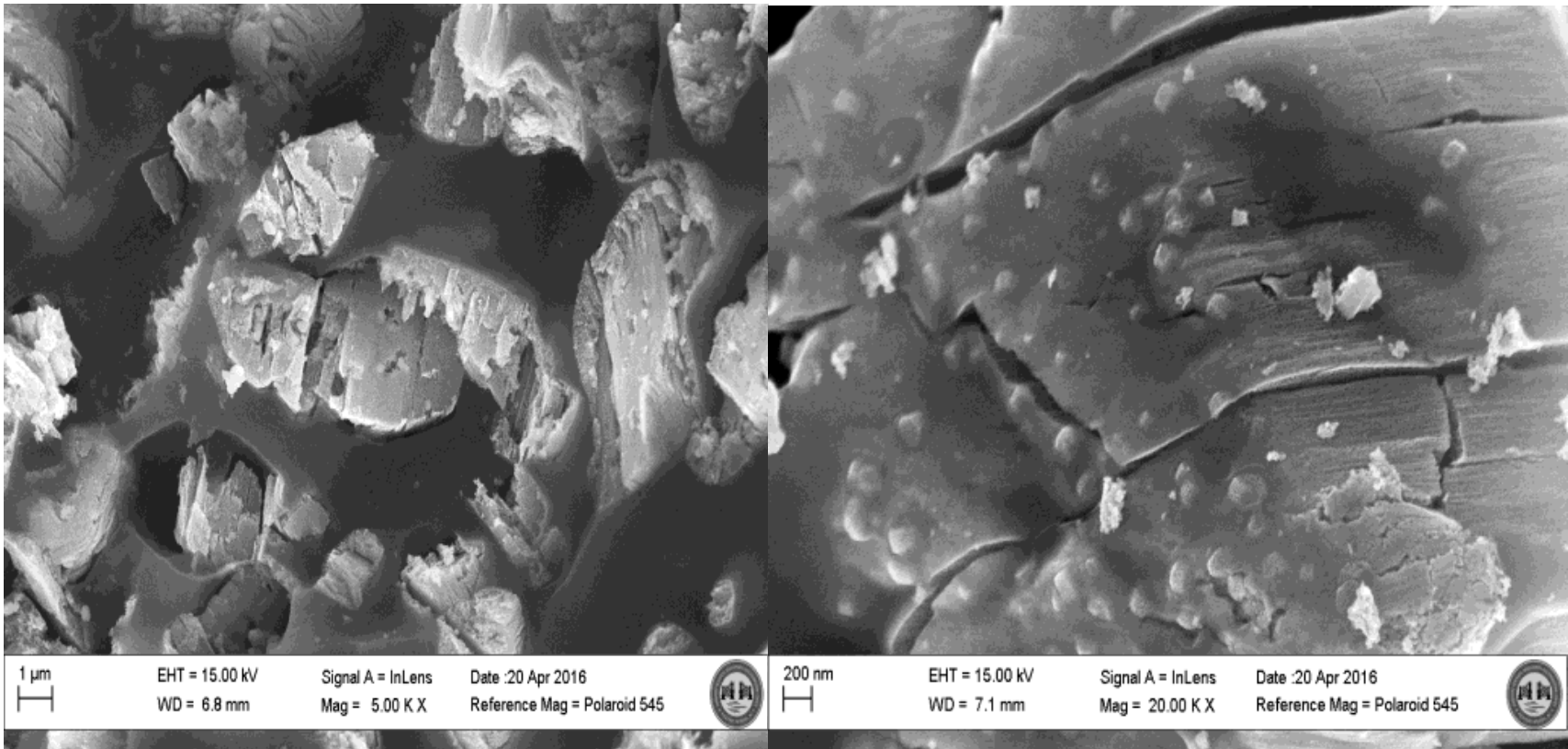
# RESULTS



## Foam materials produced at 500 °C in 5% hydrogen in Argon

*Cured*

*Activated*



**Figure 6.** Scanning electron microscopic (SEM) images of the pyrophoric foam materials following thermal treatment in Argon at 500 °C.



## CONCLUSIONS



### A NOVEL MATERIAL:

Disclosed is highly pyrophoric foam materials, which contain

- Highly dispersed nanosized Fe<sup>0</sup> particles in a custom-built carbon foam matrix.
- Both the nanosized Fe<sup>0</sup> particles and the carbon foam matrix are flammable upon exposure to the air.
- High moldability of the precursor formulation prior to further thermal activation
- Possibility of fabricating pyrophoric foams with any desirable geometry:
  - Thin disks, beads, granules
  - Monoliths with channels and cavities
  - Pellets in various shapes, etc.
  - w/ broader applications

### + AN INNOVATIVE PROCESS:

The pyrophoric foam materials are made via a simple one-pot synthesis process.

An iron-containing compound which releases gases upon heating, such as iron oxalate (Fe (CO<sub>2</sub>)<sub>2</sub>•2H<sub>2</sub>O), is used as the preferred iron precursor.

A thermoset polymer, such as resole phenolic resin, is used as the preferred carbon precursor.

Formation of nanosized Fe<sup>0</sup> particles and carbon foam matrix occur concurrently.

Therefore, the synergy between these chemical reactions is fully exploited.

Superb flexibility of the process for desirable exothermic characteristics of the pyrophoric foam materials.