



# Effect of Microstructure Control on Reaction Characteristics in Al/Ni Reactive Powder

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# Outline

- Background and Objectives

(what is reactive materials (RM) and reactive material structures (RMS))

- Experimental Procedure

(how to make RM and RMS)

- Microstructure Characteristics

- Reaction Characteristics

- Summary

# Reactive Material (RM)

- **Reactive materials (RM)** are mixtures such as metal-metal, metal-oxide, and metal-polymer that cannot be detonated, but are **capable of releasing large amounts of thermodynamic energy** very rapidly.
- Examples of RM

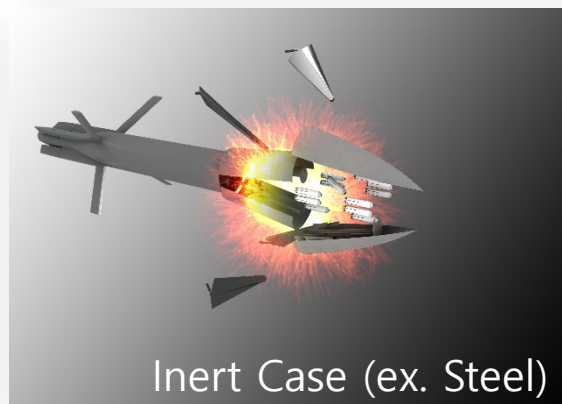
※ TNT : 1,900 cal/cc

Category	System	Heat of Reaction (cal/cc)
<b>Metal-metal (Intermetallic)</b>	<b>Al+Ni=AlNi</b>	<b>1,710</b>
	<b>Al+Ni=AlNi + O<sub>2</sub> = NiAlO<sub>2</sub></b>	<b>8,000</b>
	<b>2Al+Zr=Al<sub>2</sub>Zr</b>	<b>1,130</b>
	<b>2Al+Ti=Al<sub>2</sub>Ti</b>	<b>1,100</b>
<b>Metal-oxide (Thermite)</b>	<b>2Al+3CuO=3Cu+Al<sub>2</sub>O<sub>3</sub></b>	<b>4,976</b>
	<b>2Al+Fe<sub>2</sub>O<sub>3</sub>=2Fe+Al<sub>2</sub>O<sub>3</sub></b>	<b>3,947</b>
<b>Metal-polymer</b>	<b>Al-PTFE</b>	<b>6,000</b>
<b>Metal-non metal</b>	<b>3Ti-5Si=Ti<sub>3</sub>Si<sub>5</sub></b>	<b>428</b>

- **RMS**, which are made of RM powder, are **energetic structures** designed to have structural strength and store energy to be released at a desired time.

# Reactive Case

- Inert structural materials based on steel are normally used as missile's cases
- If we **replace the inert steel case** currently used **with reactive material structures**?

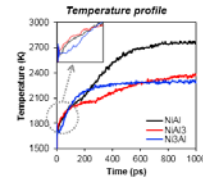
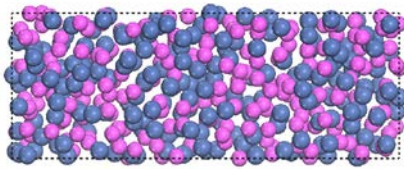


- The delivered energy to target would be increased, because **the RMS case is capable to react in the exploding environment of explosives**, unlike steel case
- ADD has been working on a project to develop reactive cases since 2014

# Objectives

- Secure the core technologies to develop reactive cases

## Design technology



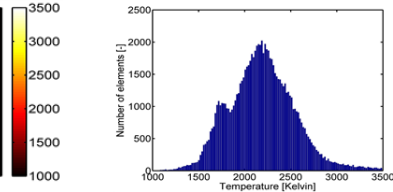
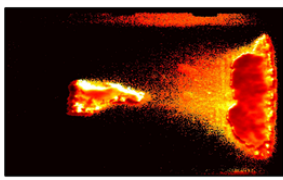
- Design technology to design RMS that release a large amount of energy

## Fabrication technology



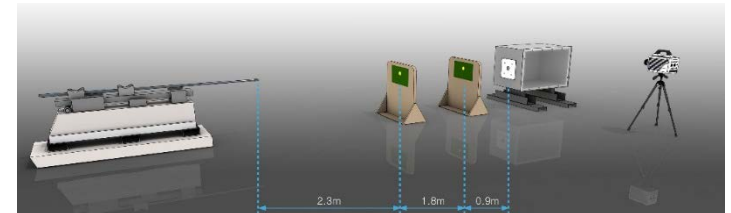
- Fabrication technology to fabricate RM powder and RMS as designed states

## Analysis technology



- Analysis technology to analyze acquired data from RMS tests

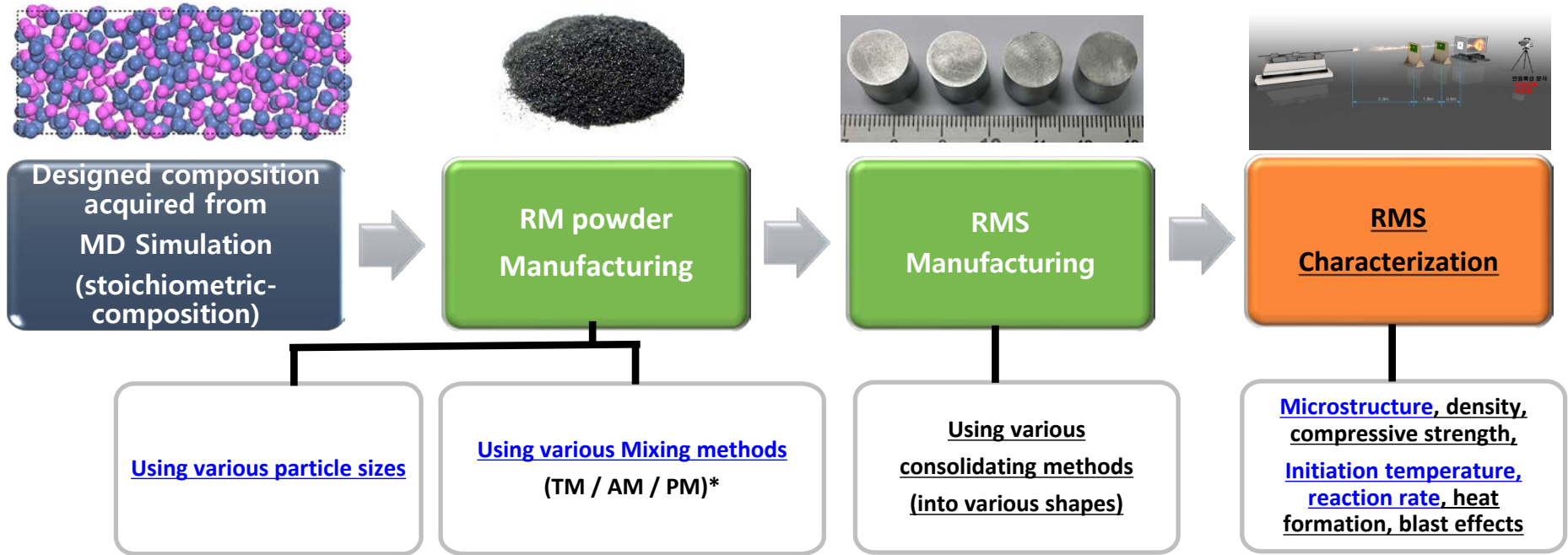
## Test technology



- Test technology to simulate the RMS operating environment

# Schematic Procedure for developing RMS

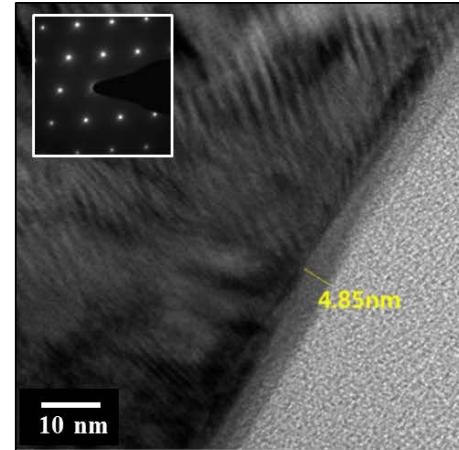
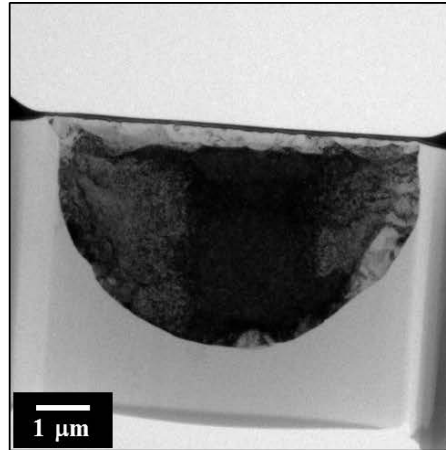
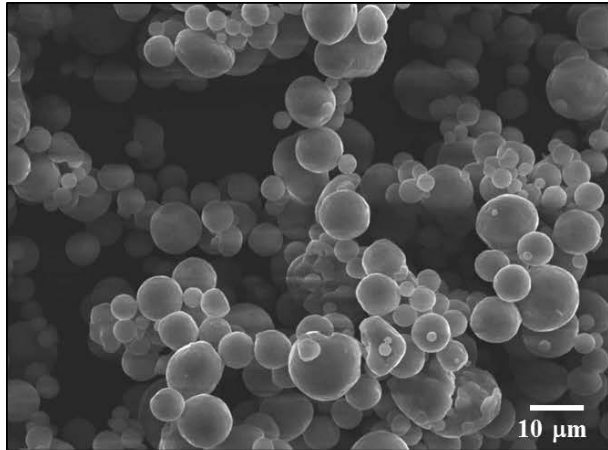
\*TM: Turbula Mixing // AM: Attrition Milling // PM: Planetary Milling



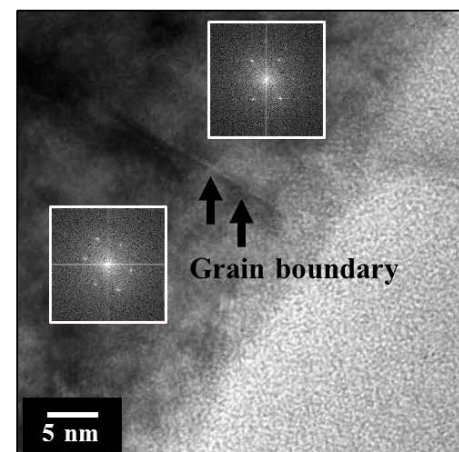
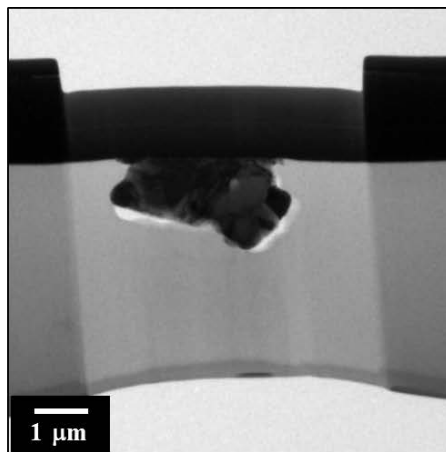
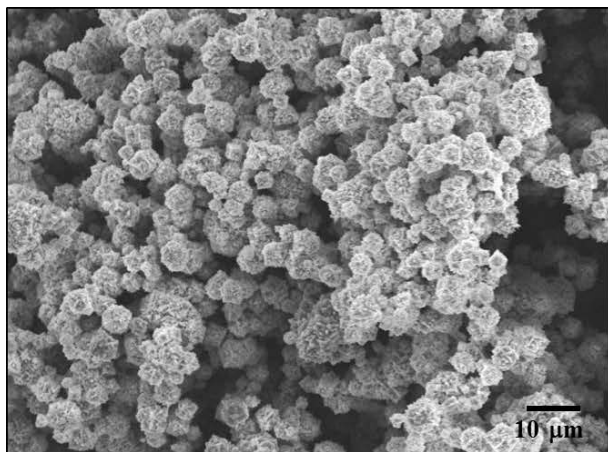
- How to make RM powder
- Microstructural development according to mixing methods
- A Correlation between the microstructure and their reaction characteristics

# Microstructure of raw powder (Al, Ni)

## ■ Spherical shape of Al (AVG = 10 $\mu\text{m}$ )



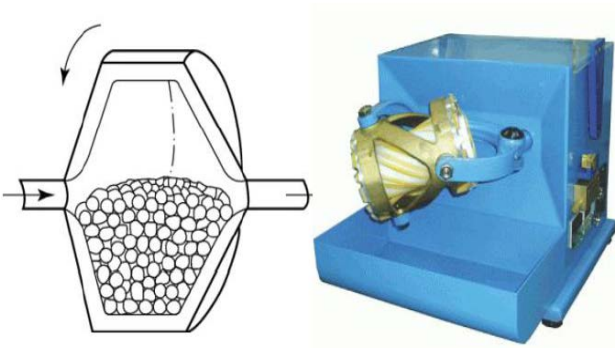
## ■ Needle (spiky) shape of Ni (AVG = 4 $\mu\text{m}$ )



# Manufacturing of Al/Ni reactive powder

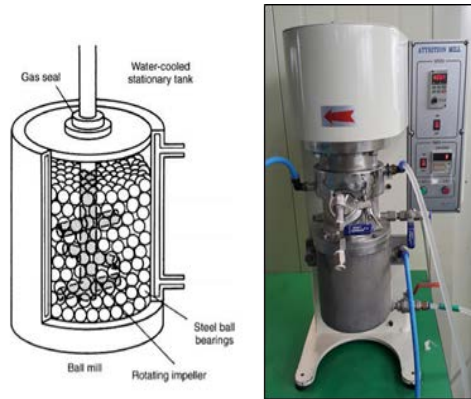
■ Use 3 types of mixing methods to modulate microstructure of Al/Ni reactive powder

## ■ Turbula Mixing (TM)



➔ 3D movement of container

## ■ Attrition Milling (AM)



➔ Rotation of impeller in container

## ■ Planetary Milling (PM)

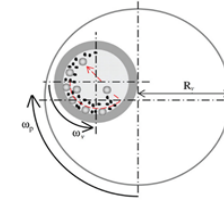


Figure 1. The schematic diagram of the planetary ball mill and the visit.



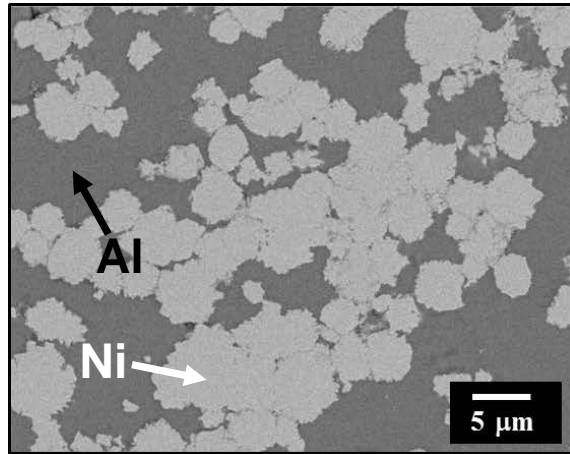
➔ Planetary (rotation and revolution) movement of container

➤ The amount of energy applied to the powder gradually increases in order of TM, AM, and PM.

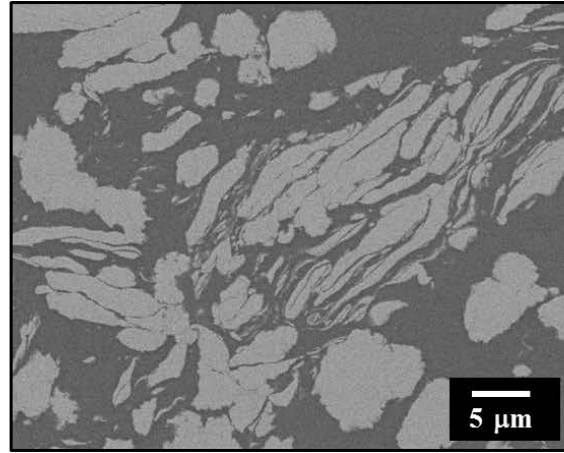


# Microstructure of the Al/Ni reactive powder

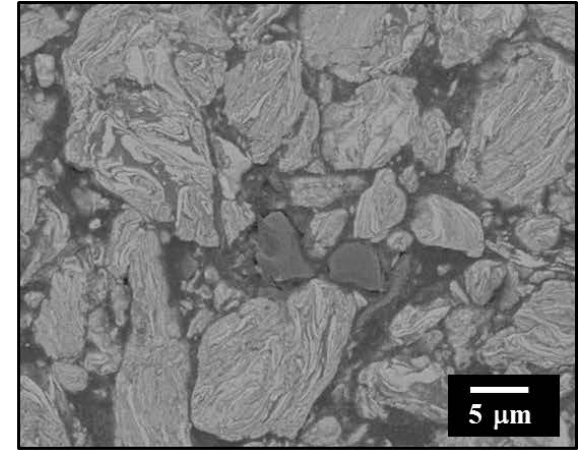
## ■ Turbula Mixing (TM)



## ■ Attrition Milling (AM)



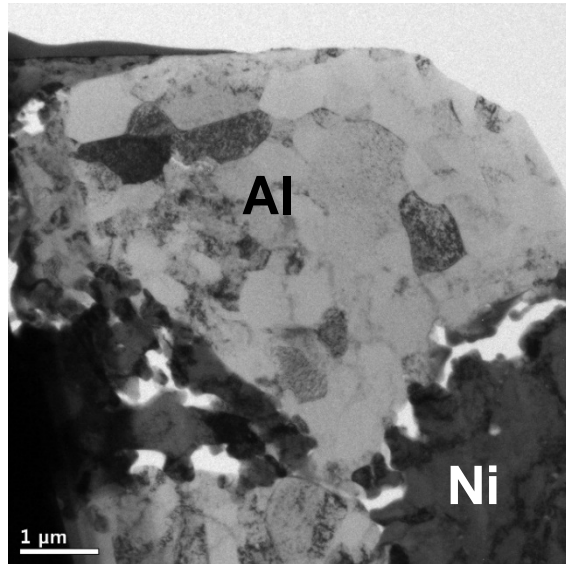
## ■ Planetary Milling (PM)



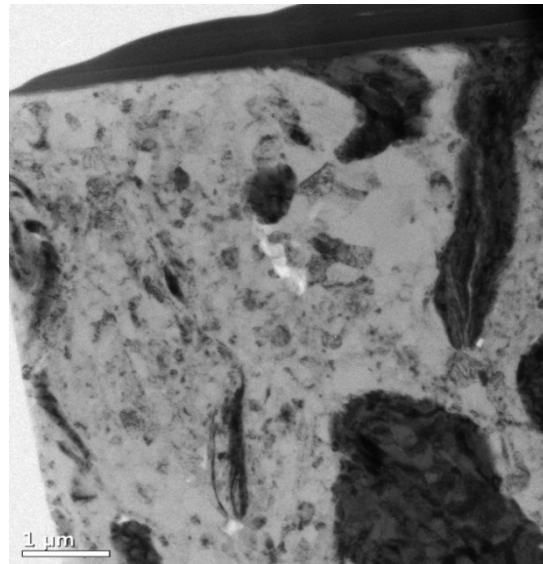
- In TM-powder, the original shape of Al and Ni raw powder is maintained
- In AM-powder, the needle shape of Ni gradually disappear and shows elongated microstructure
- In PM-powder, the original shape of raw powder is completely collapsed and mixed at nano-level

# Microstructure of the Al/Ni reactive powder

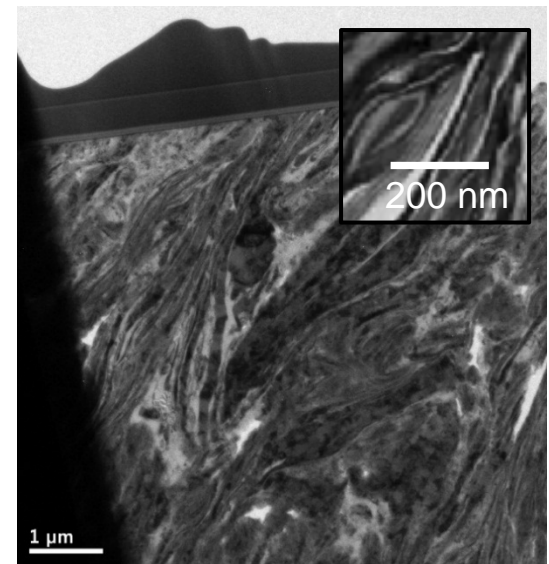
## ■ Turbula Mixing (TM)



## ■ Attrition Milling (AM)



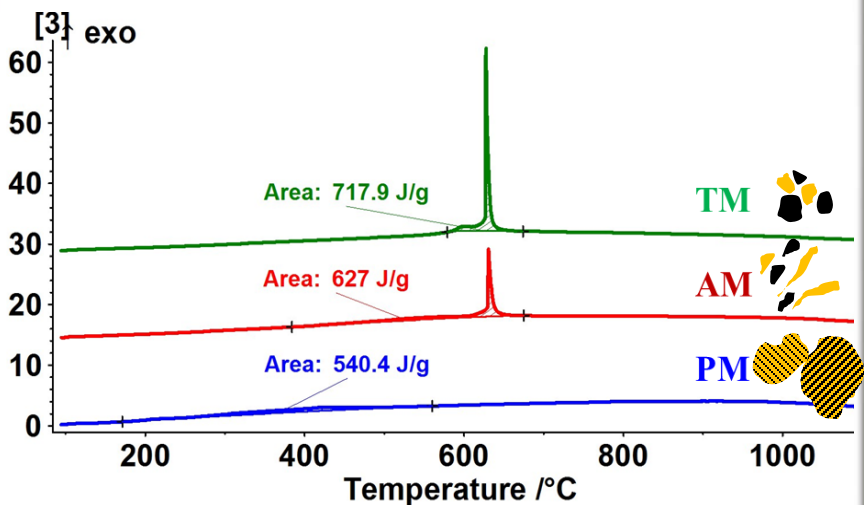
## ■ Planetary Milling (PM)



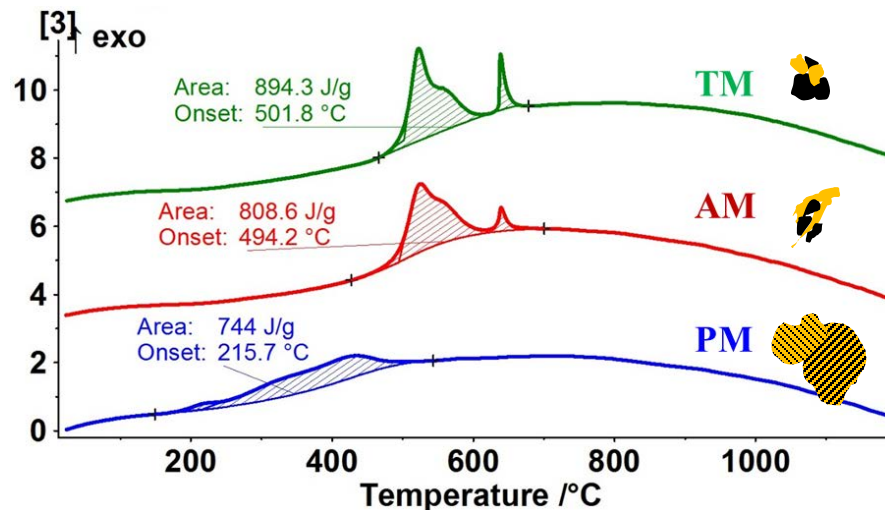
- In TM-powder, the needle shape of Ni is maintained, but Al particle is broken into several grains of 1  $\mu\text{m}$  (grain refinement of Al particle)
- In AM-powder, the grain refinements are more pronounced, and most of Al grains represent sub-micron size
- In PM-powder, the original shape of raw powder is completely collapsed and shows nano-lamella structure in which Al layer and Ni layer stacked alternatively and mixed at nano-level

# DSC analysis

## Powder DSC Curve



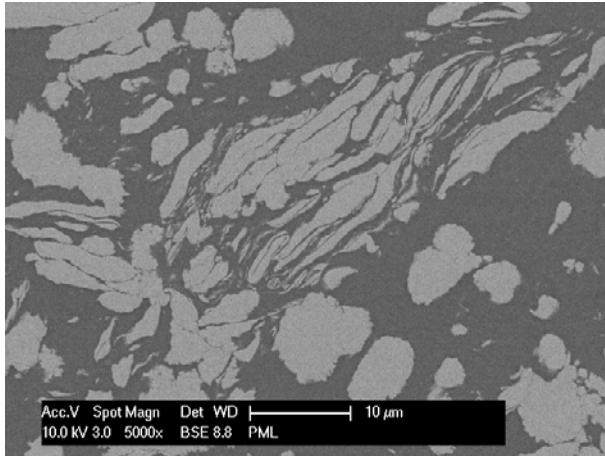
## Compact DSC Curve



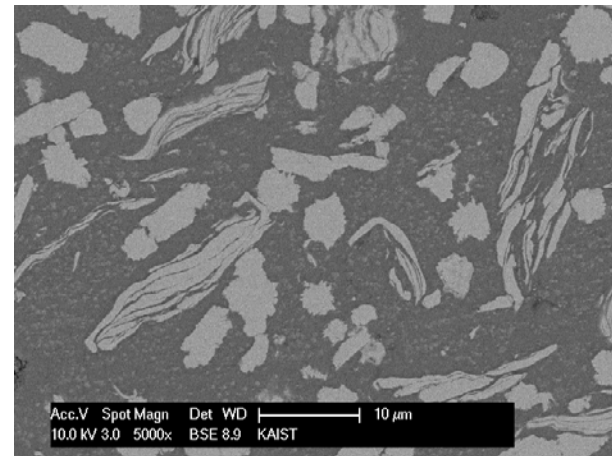
- ➔ TM- and AM- powder are reacted around Al melting point which is about 660 °C (hetero. Rx)
- ➔ PM-powder is reacted below Al melting point => PM-powder can react in solid state (homo. Rx)
- ➔ TM- and AM- compact are reacted at about 500 °C (the reaction initiation temperature changes)  
=> In order for the reaction to initiate, it is important whether the interface between components is bonded or not
- ➔ The quantity of heat of reaction is gradually decreased with microstructure development  
=> A small amount of components are already reacted in the mixing process.

# Microstructure of the Al/Ni reactive powder

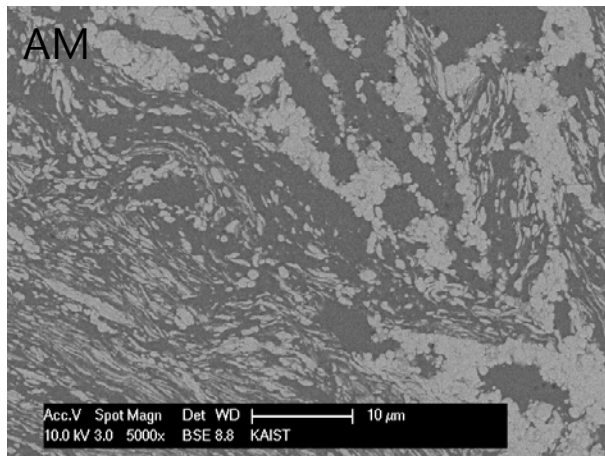
■ Al (10  $\mu\text{m}$ ) + Ni (4  $\mu\text{m}$ ) -



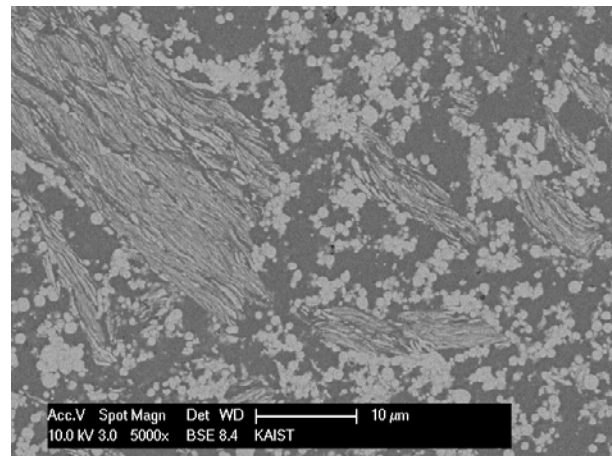
■ Al (1  $\mu\text{m}$ ) + Ni (4  $\mu\text{m}$ ) - AM



■ Al (10  $\mu\text{m}$ ) + Ni (1  $\mu\text{m}$ ) -

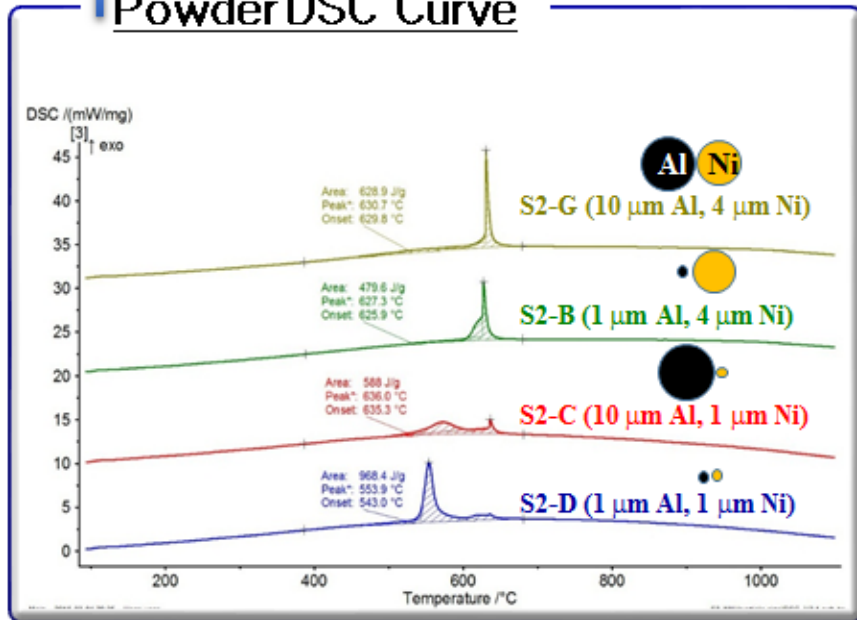


■ Al (1  $\mu\text{m}$ ) + Ni (1  $\mu\text{m}$ ) - AM

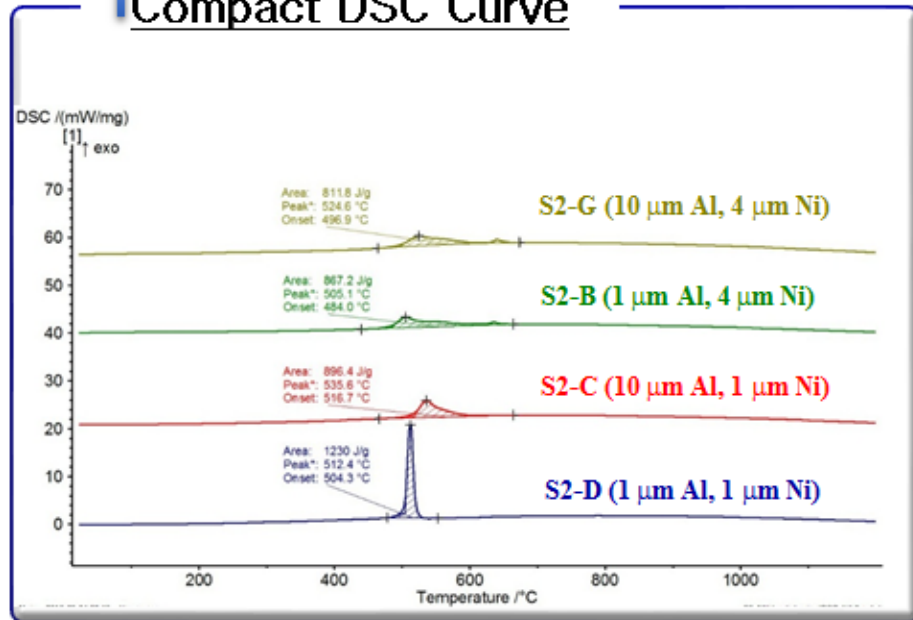


# DSC analysis

## Powder DSC Curve



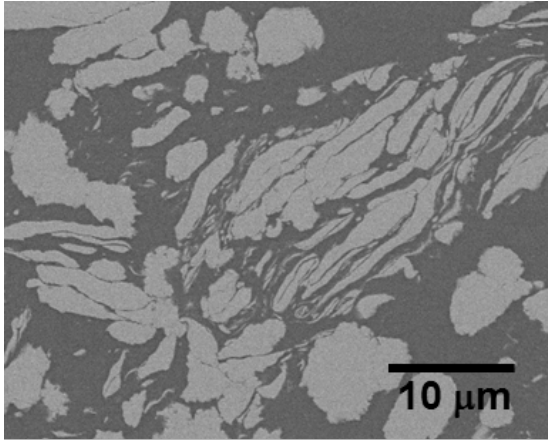
## Compact DSC Curve



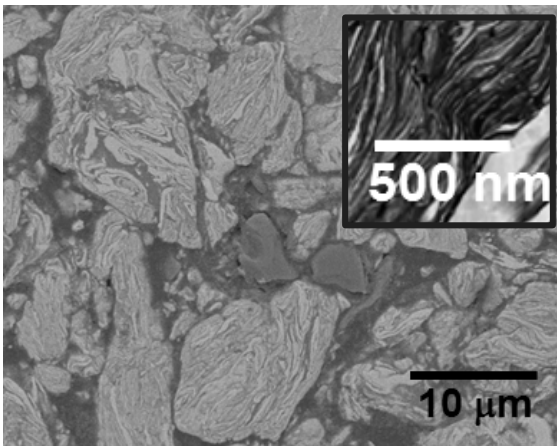
- ➔ The reaction initiation temperature is dropt only when using 1  $\mu\text{m}$  Ni powder  
=> In order for a RM to react, the mass transport of the component exhibiting a slow diffusion rate becomes important
- ➔ In the case of compact analysis, there are no big differences in terms of reaction initiation temperature  
=> In order for the reaction to initiate, it is important whether the interface between components is bonded or not

# Reaction rate (of RM compacts with changing microstructure)

- Attrition Milling (AM): The reaction is completed within about 100 ms to propagate 5 mm (0.05 m/s)



- Planetary Milling (PM): The reaction is completed within about 10 ms to propagate 5 mm (0.5 m/s)



# Summary

- Various types of Al/Ni RM powders were prepared by varying mixing methods, and they are clearly distinguishable in terms of microstructure
- In DSC analysis, the reaction initiation temperatures of RM powder and RM compacts varied more than 200 °C with the changes of microstructure
- In order for the reaction to initiate, it is important whether the interface between components is bonded or not
- The reaction propagation rate varies greatly depending on the microstructure
- ➔ The reaction characteristics of the RM (or RMS) could be controlled by tailoring the microstructure of RM (or RMS)



Thank You

Q & A