

Structural, morphological and magnetic characteristics of Aluminium Superalloy new magnetic material obtained by mechanical alloying

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F. Popa, C.V. Prică, I. Chicinaș

Why Supermalloy based materials?

Supermalloy - Ni75Fe20Mo5

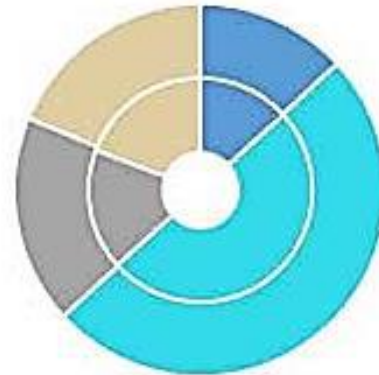
- Good magnetic induction, extremely high magnetic permeability (1000000), and a low coercivity
- Supermalloy is very useful in ultra-sensitive transformers, especially pulse transformers, and ultra-sensitive magnetic amplifiers where low loss is mandatory.

Global Soft Magnetic Composites Market Value Share, by Region



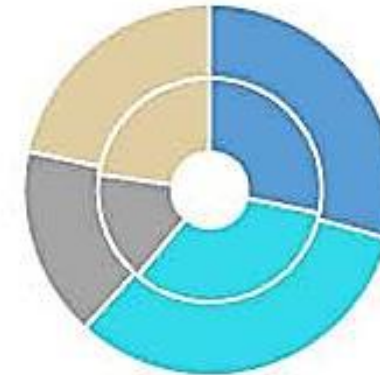
North America Latin America
Europe Japan
APEJ MEA

Global Soft Magnetic Composites Market Value Share, by Product Type



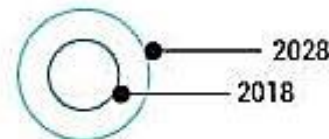
Pure Iron/Iron Powder Silicon Ferrite
Supermalloy Permalloy

Global Soft Magnetic Composites Market Value Share, by Application



Transformers Motors
Inductors Generators

Source: Fact.MR

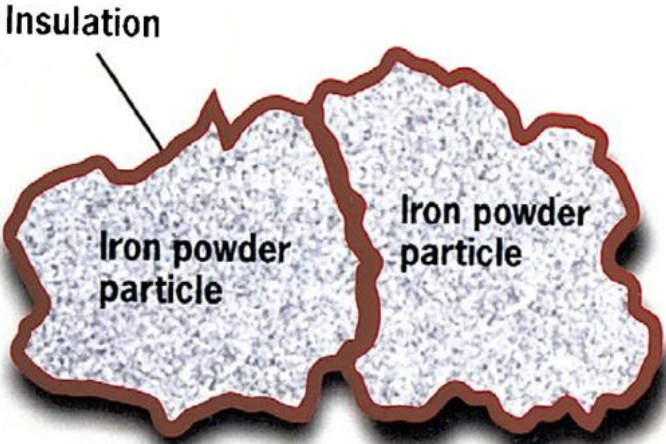


Fact.MR

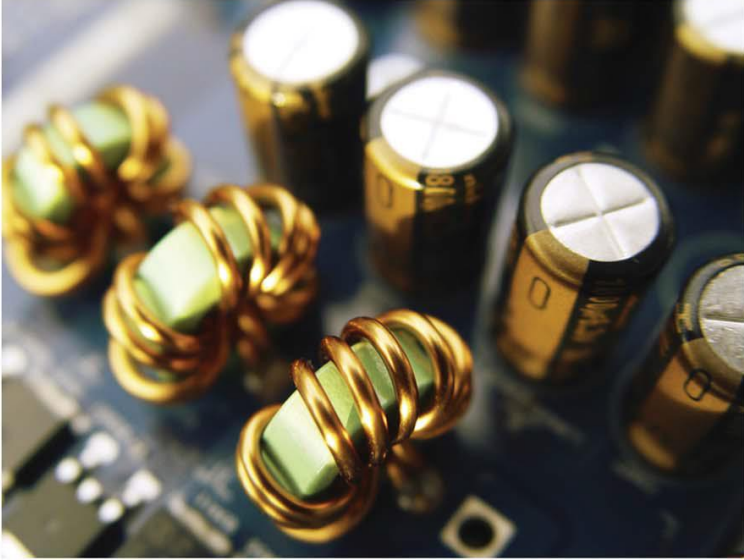
Soft magnetic composite (SMC) market value share analysis

Research Conference of Technical University of Cluj-Napoca, October 20-22, 2021

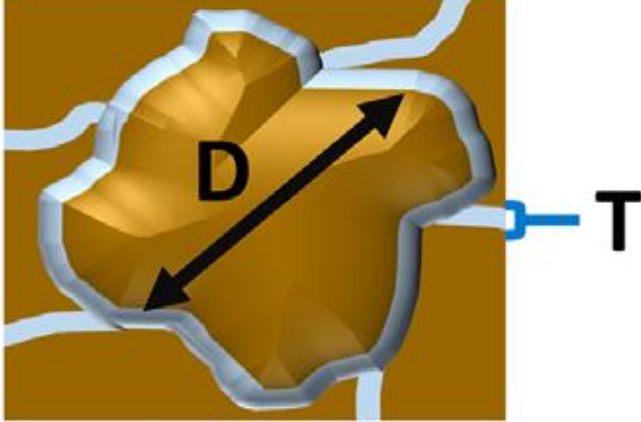
Why soft magnetic composite material



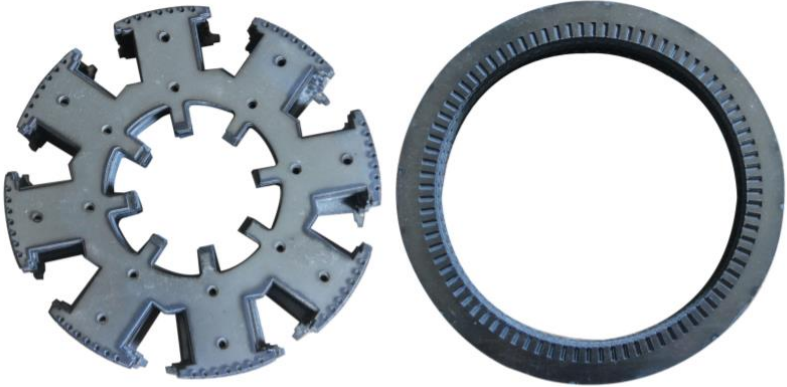
A schematic diagram of the component elements of a powder core.



Examples of toroidal inductors on printed circuit board – PCB

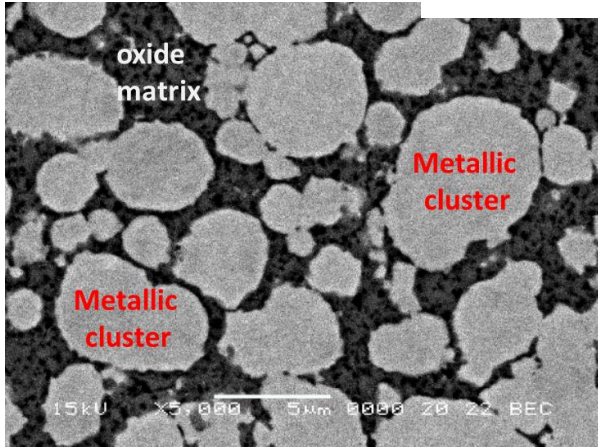
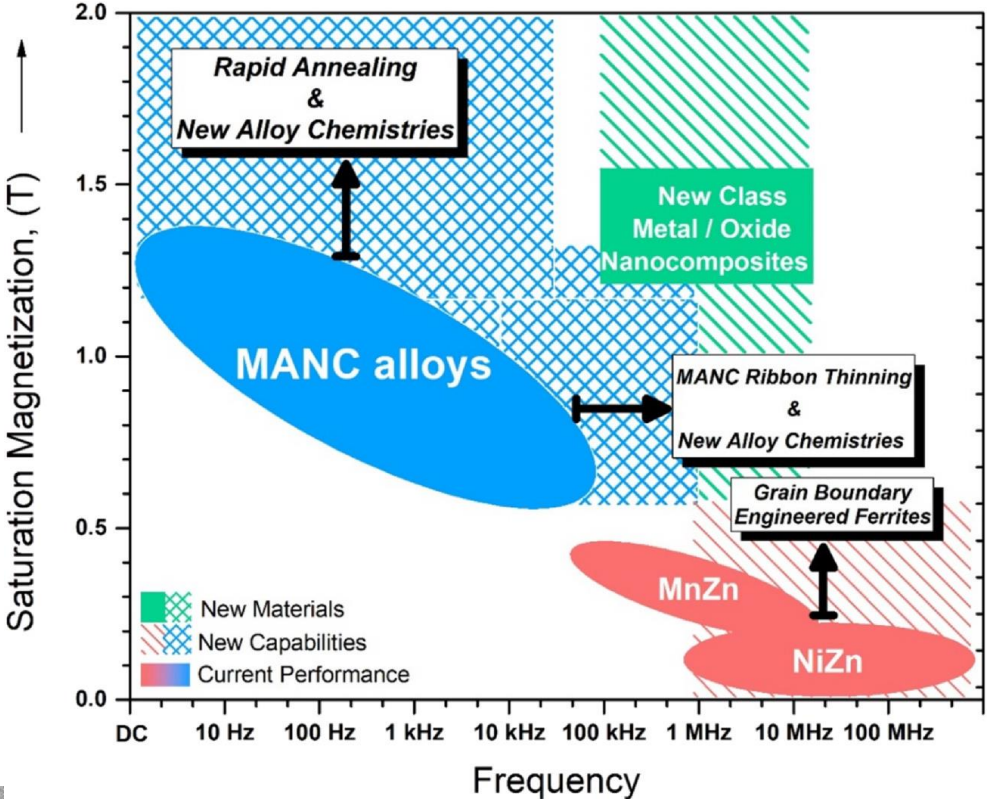
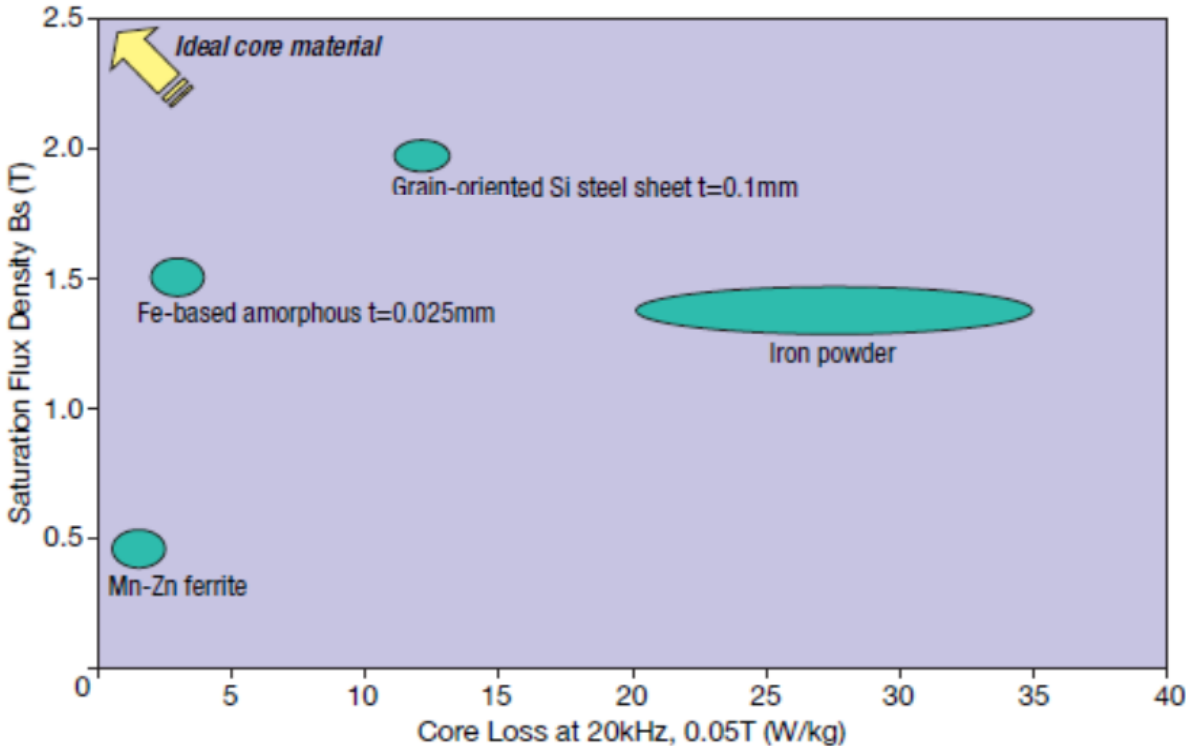


Composite after pressing illustrating the ferromagnetic part and the insulating layer.

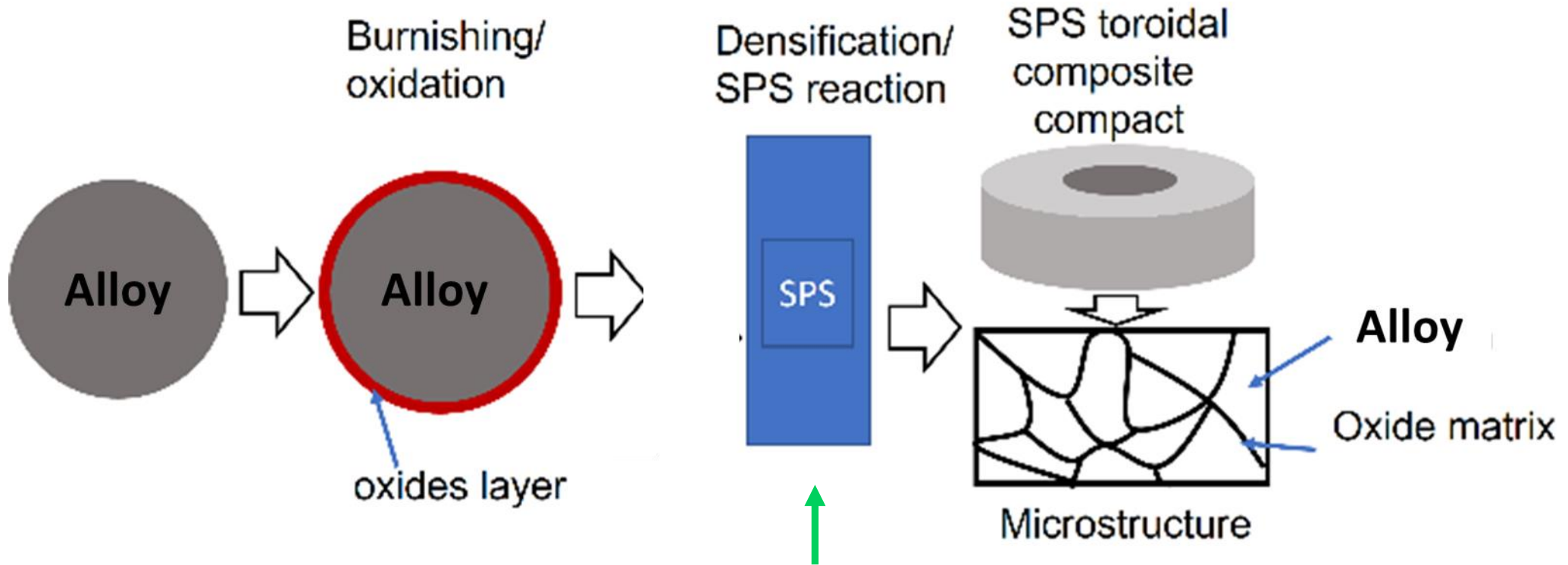
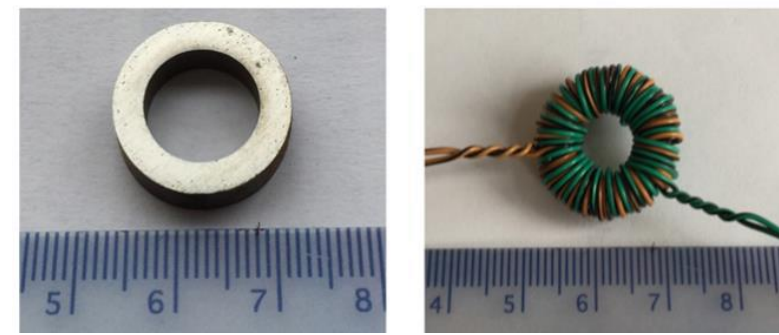


Parts made from supermalloy for motors and generators

Why composite Supermalloy based@oxide



Why Aluminium Superalloy?



During SPS reaction between alloy core and oxide shell

Experimental

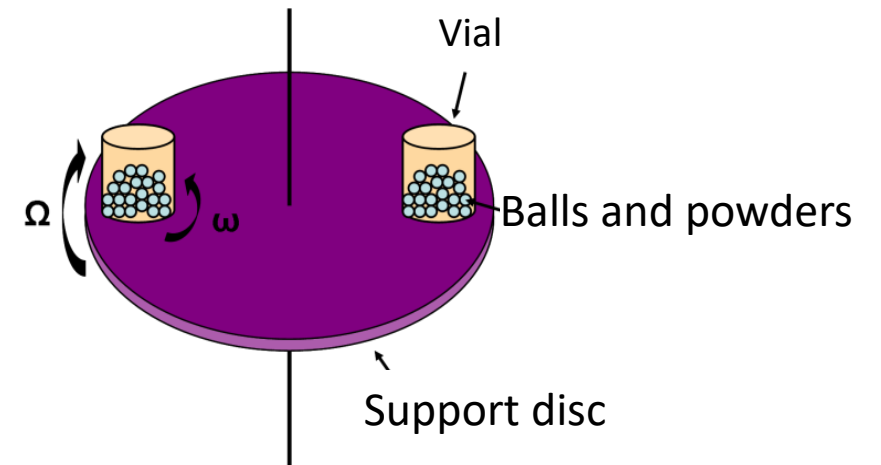
$\text{Ni}_{70.5}\text{Fe}_{18.8}\text{Mo}_{4.7}\text{Al}_6 = [\text{Superalloy (Ni}_{75}\text{Fe}_{20}\text{Mo}_5)]_{94} + \text{Al}_6$ (%wt.)

$\text{Ni}_{71.25}\text{Fe}_{19}\text{Mo}_{4.75}\text{Al}_5 = [\text{Superalloy (Ni}_{75}\text{Fe}_{20}\text{Mo}_5)]_{95} + \text{Al}_5$ (%wt.)

$\text{Ni}_{71.25}\text{Fe}_{23.75}\text{Al}_5$

- Starting powders: Ni, Fe, Mo, and Al elemental powder
- Milling time: up to 20 hours
- Milling atmosphere: argon
- Sampling at: 0,5, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 15 and 20 h
- Tempered steel vials and balls: 70 balls (ϕ 14)
- Ball to powder ration (BPR): 4:1, 8:1 and 17:1
- Annealing at 450 °C for 4 h in Ar

• **Surface oxidation:** superficial powder oxidations in hydrochloric acid, in air+Ar and industrial burnishing kit



Materials characterisation

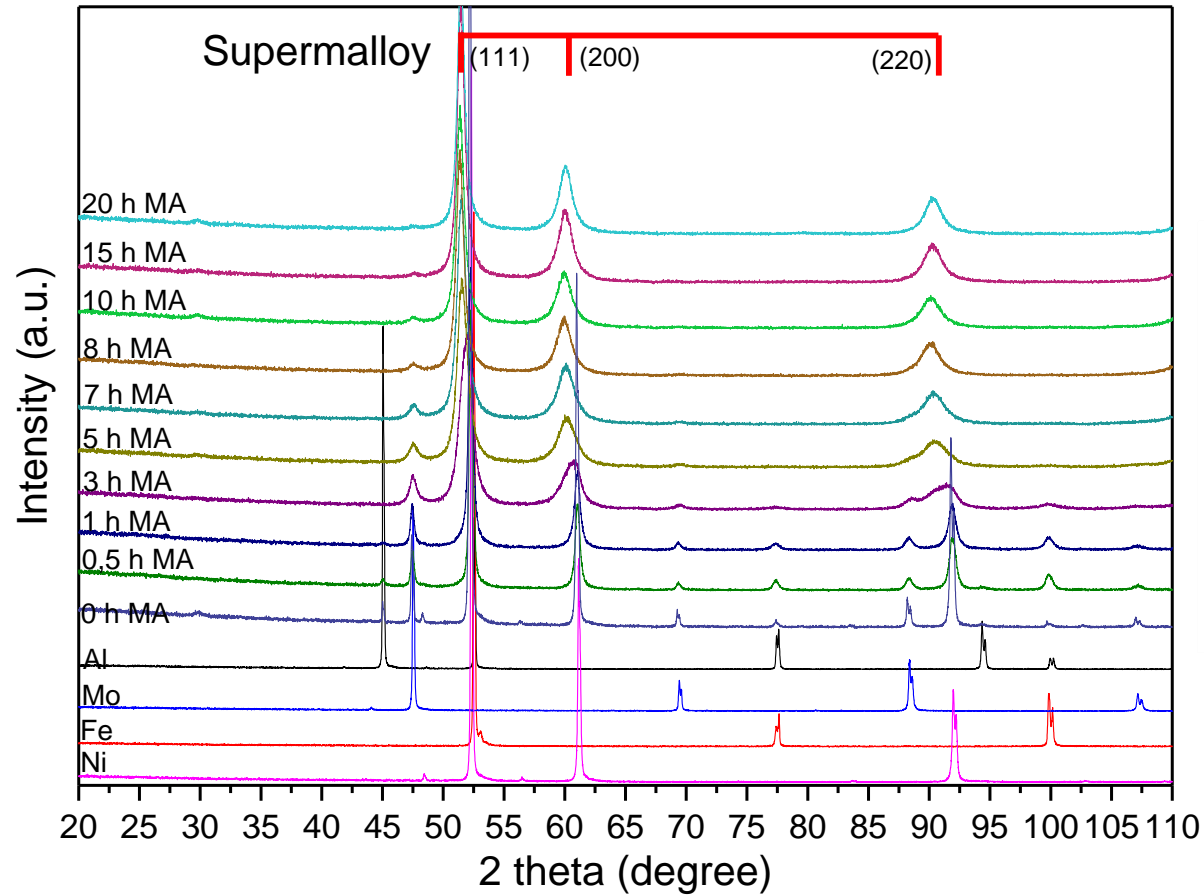
- **X-ray diffraction – XRD** – Inel EQUINOX 3000 diffractometer
- **In-situ high temperature X-ray diffraction** - Anton Paar HTK1200N heating furnace + Inel EQUINOX 3000 diffractometer
- **Scanning Electron Microscopy – SEM**: JSM 5600 LV – Jeol
- **Energy dispersive X-ray analysis - EDX**: EDX - Oxford Instruments, model ULTIMAX65, Aztec software
- **Differential Scanning Calorimetry - DSC**: Setaram Labsys apparatus
- **Fourier Infrared Spectroscopy - FTIR**: Bruker Tensor 27 Spectrometer
- **VSM magnetic measurements**



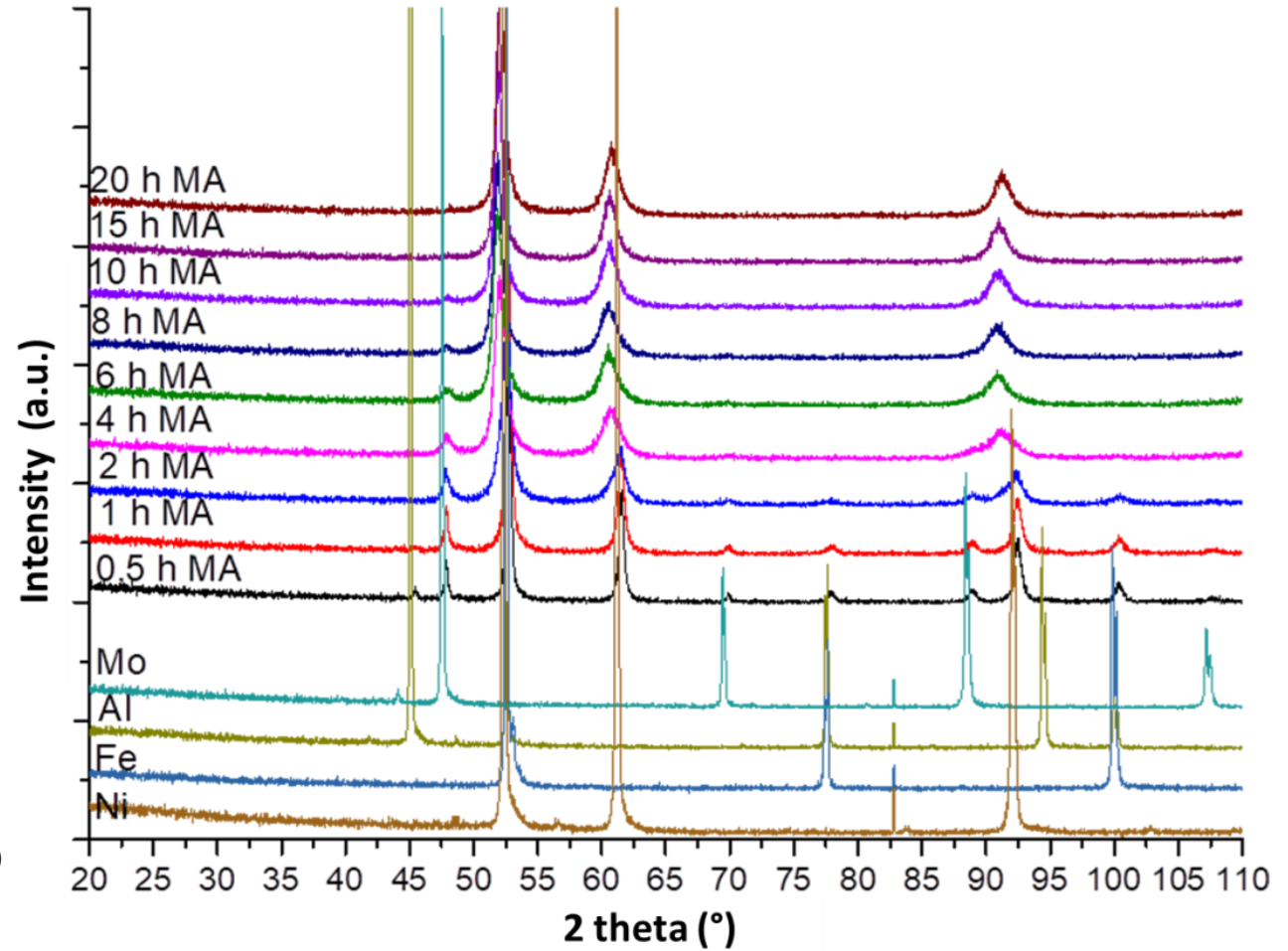
RESULTS

Formation of alloys

XRD



Ni_{70.5}Fe_{18.8}Mo_{4.7}Al₆



Ni_{71.25}Fe₁₉Mo_{4.75}Al₅

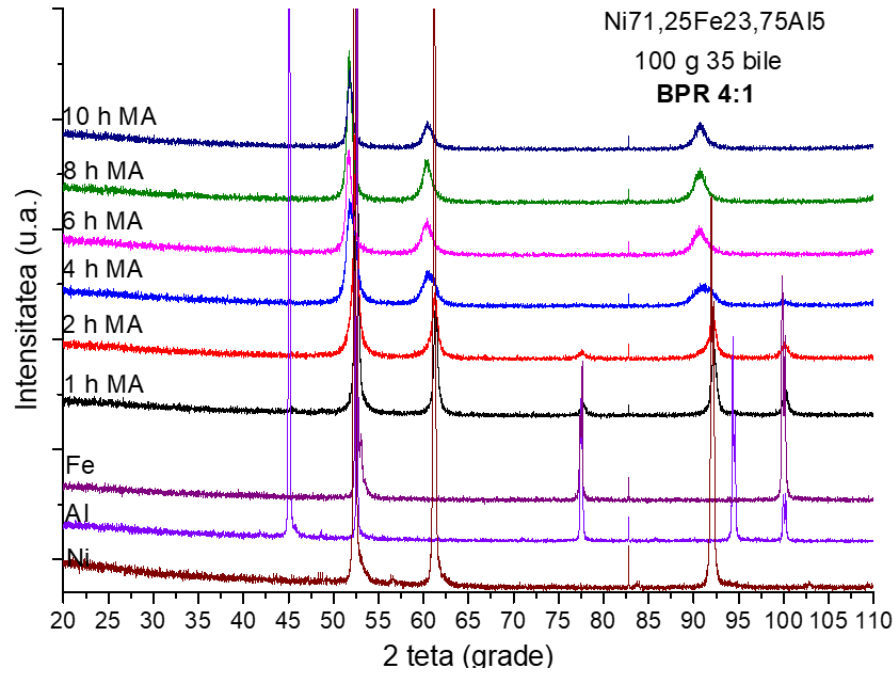
RESULTS

Formation of alloys

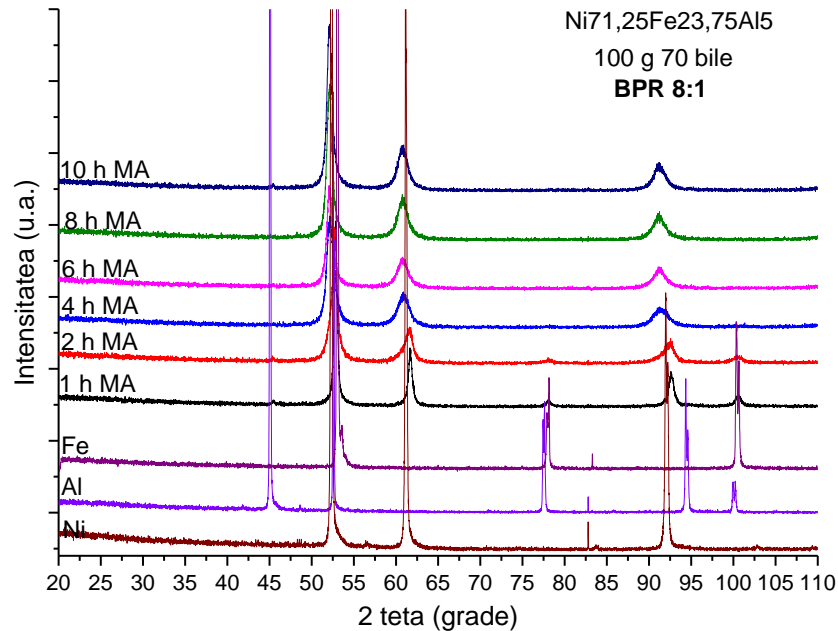
XRD

Influence of milling/alloying parameters

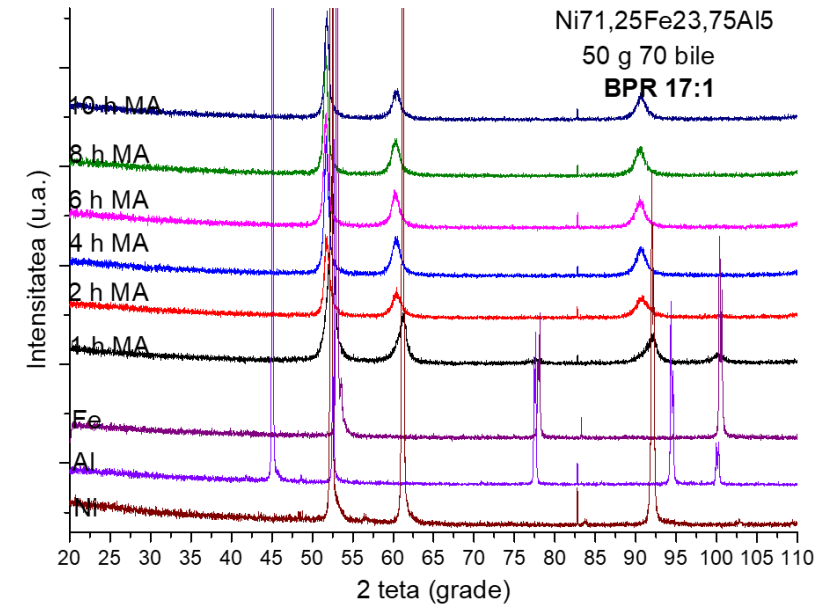
Ni_{71.25}Fe_{23.75}Al₅



Alloy obtained after 4 h



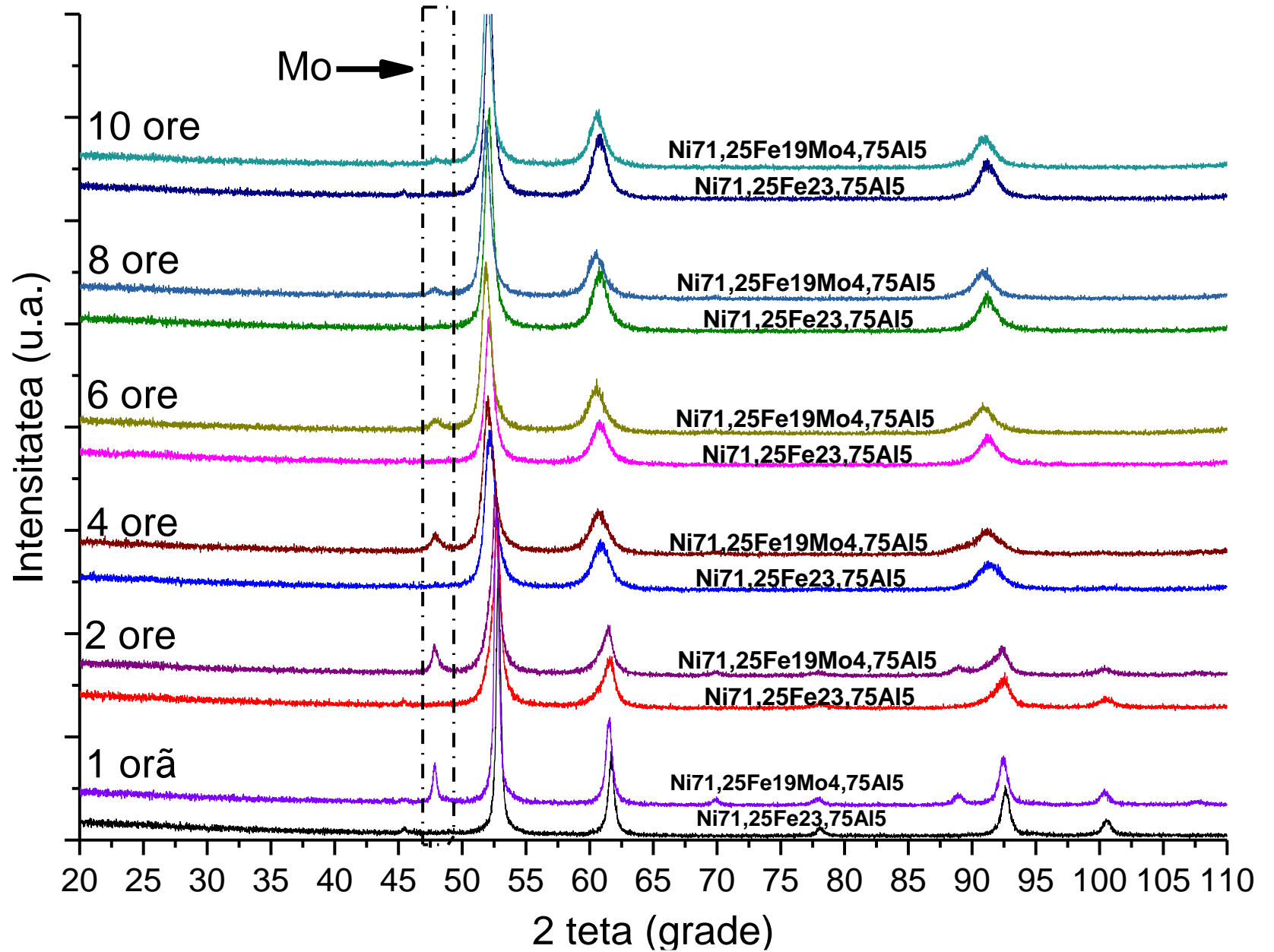
Alloy obtained after 6 h



Alloy obtained after 8 h

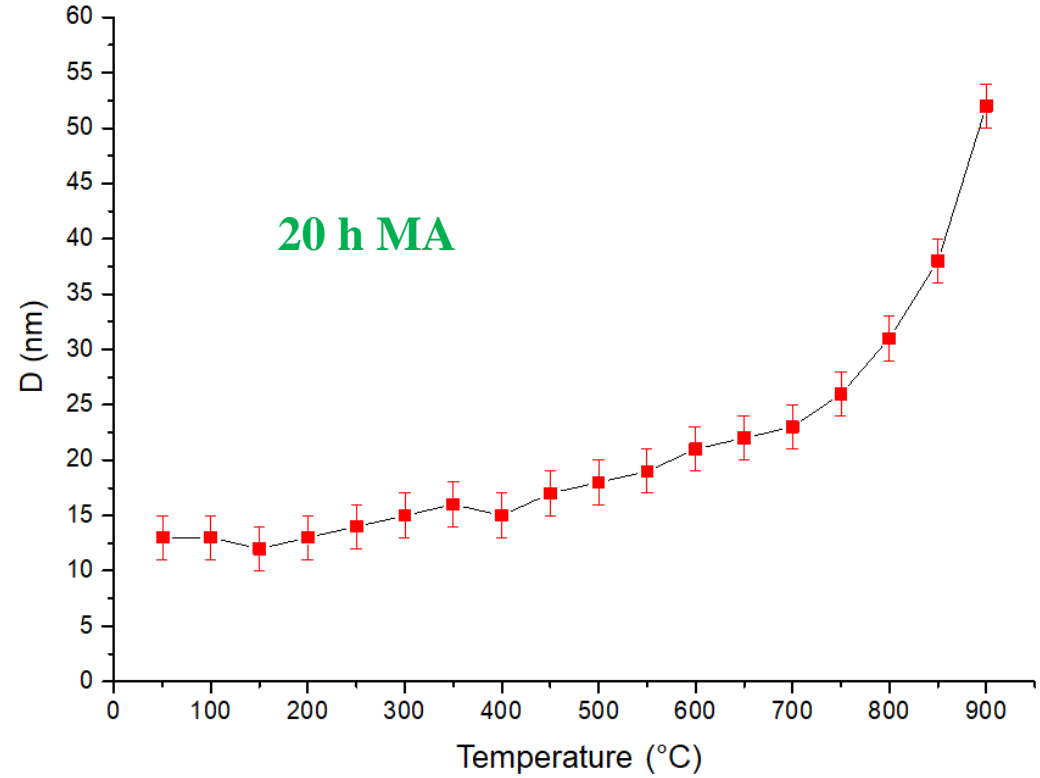
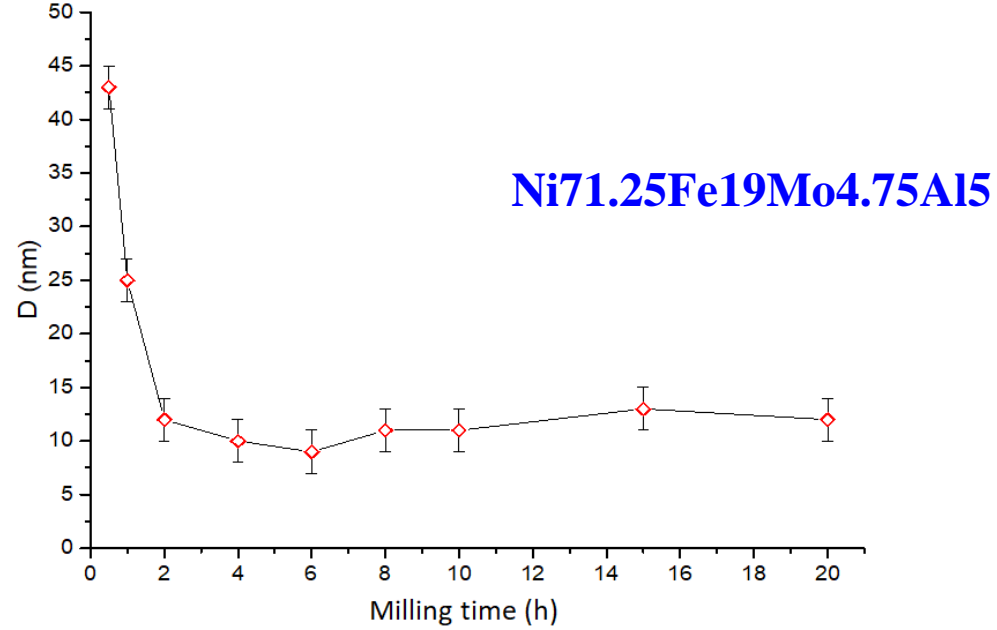
RESULTS

$\text{Ni}_{71,25}\text{Fe}_{23,75}\text{Al}_5$ and
 $\text{Ni}_{71,25}\text{Fe}_{19}\text{Mo}_{4,75}\text{Al}_5$

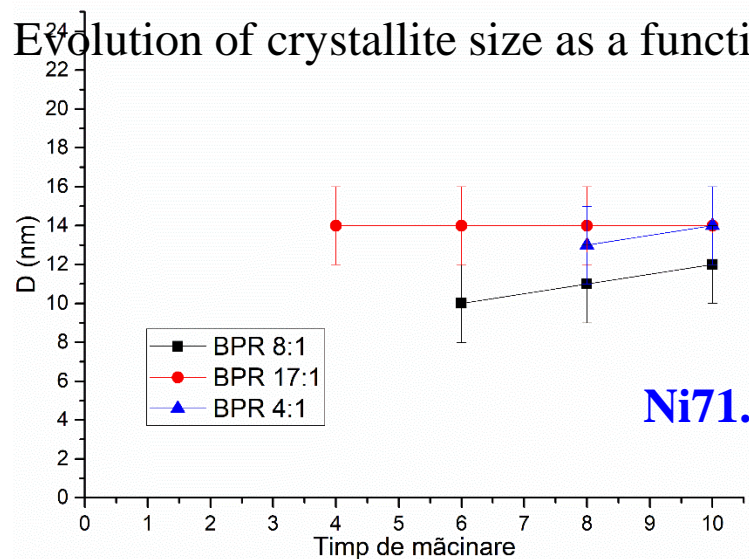


RESULTS

Crystallite size



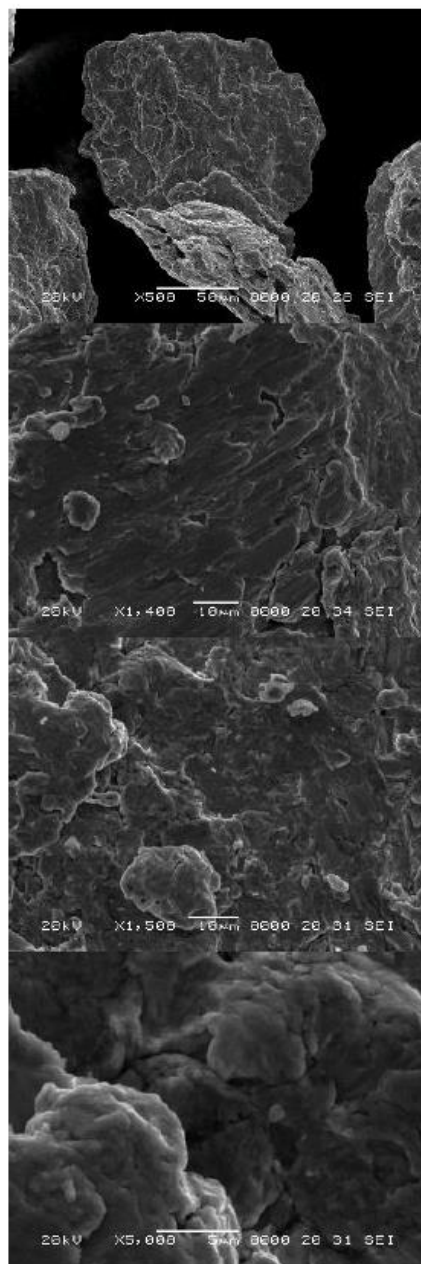
Evolution of crystallite size as a function of milling time



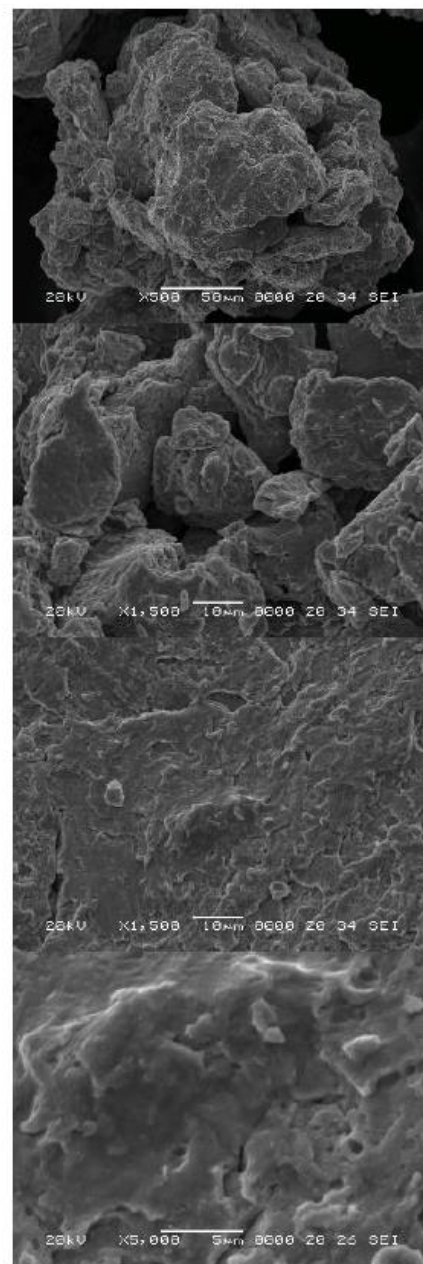
Evolution of crystallite size in temperature

SEM

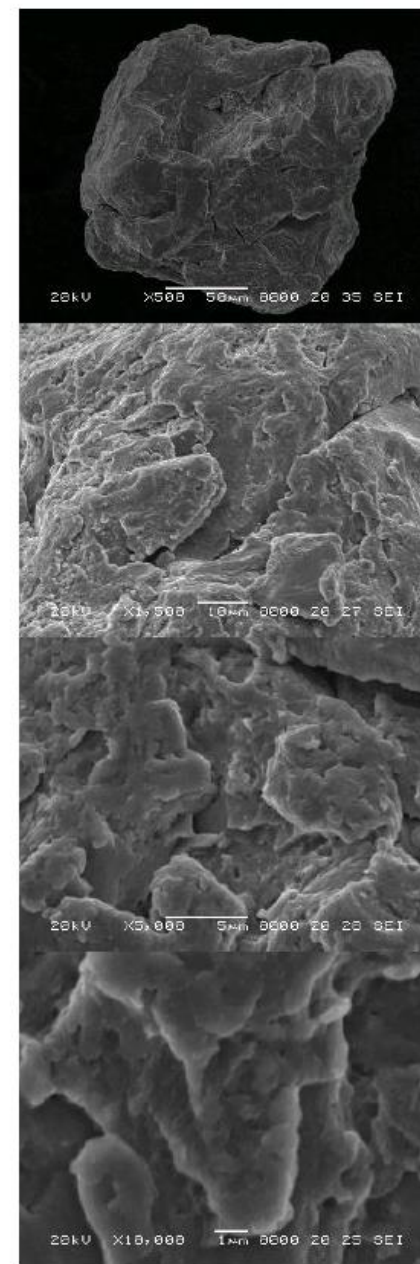
Ni70.5Fe18.8Mo4.7Al6



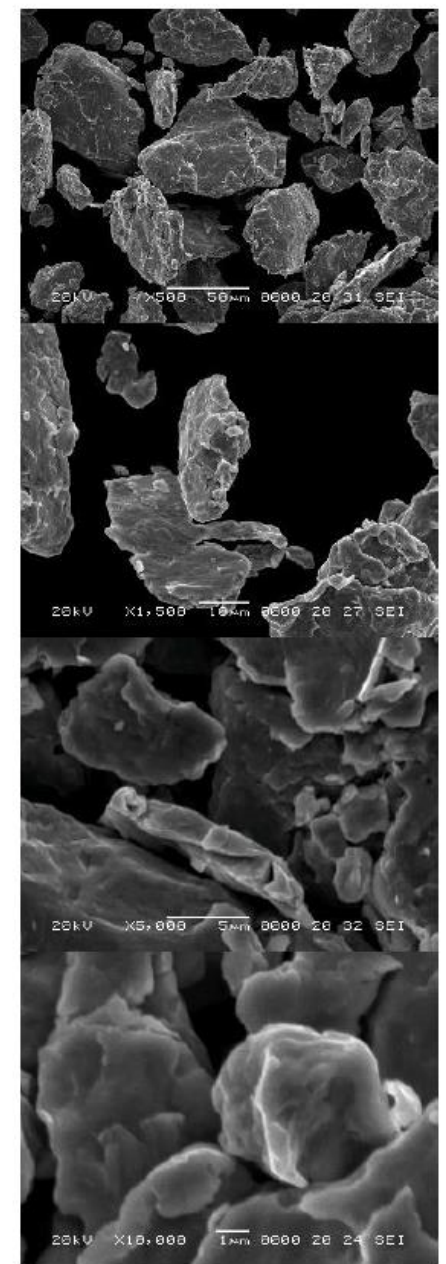
0,5 h



2 h



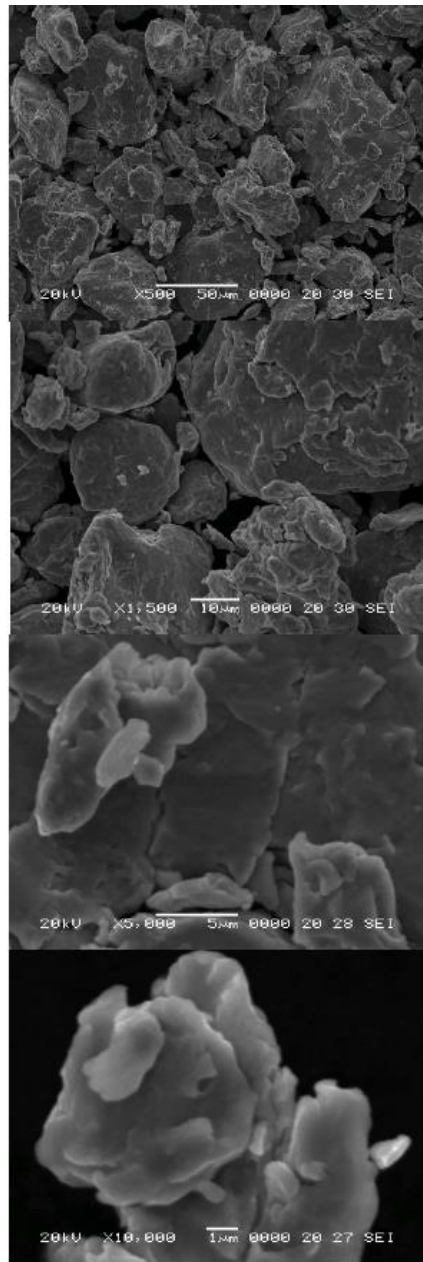
6 h



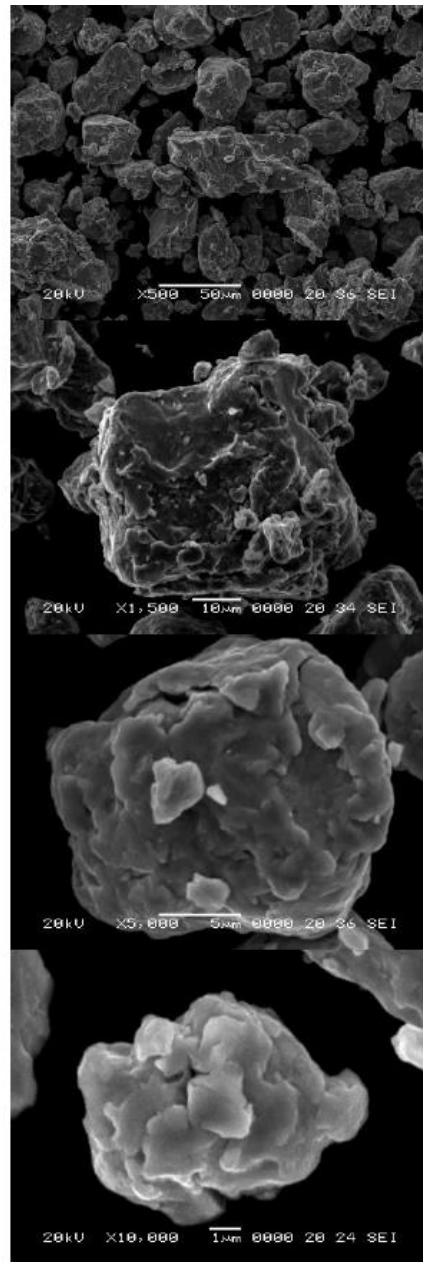
8 h

SEM

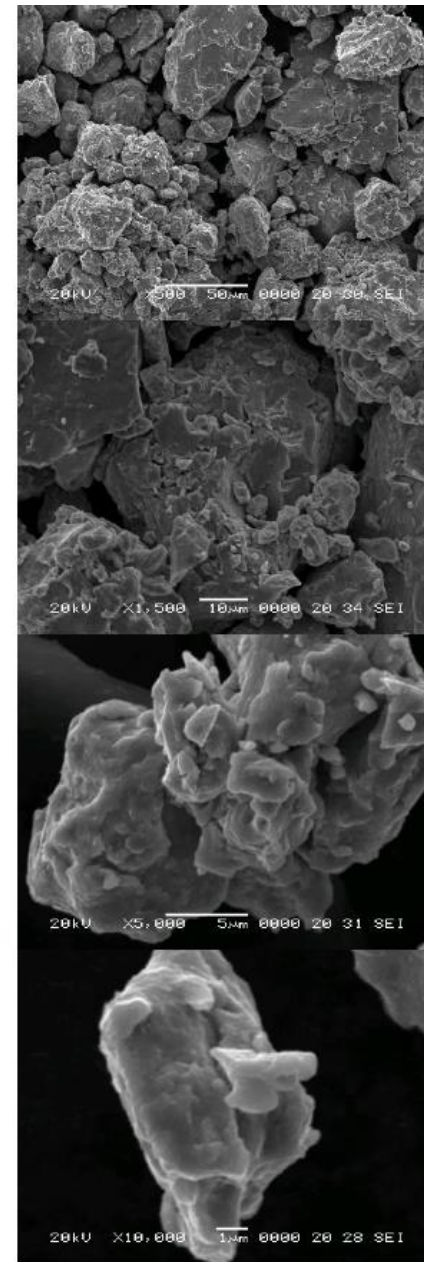
Ni70.5Fe18.8Mo4.7Al6



10 h



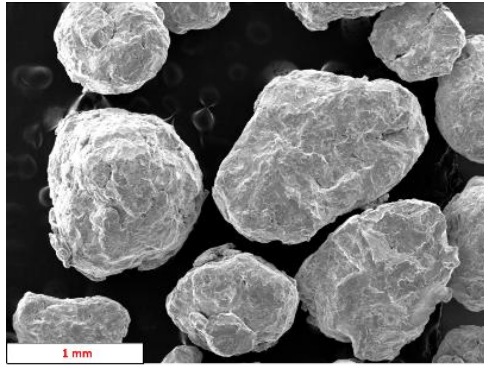
15 h



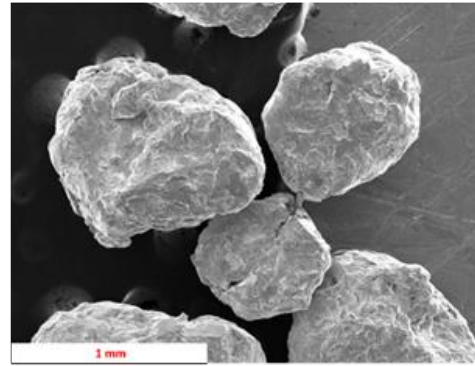
20 h

SEM

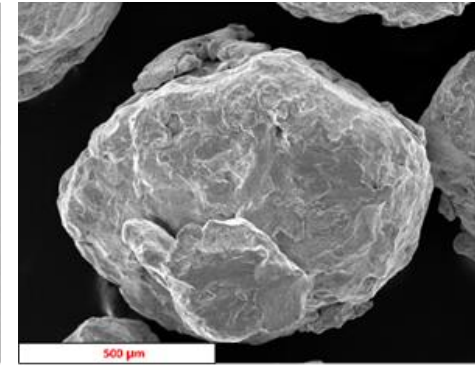
X35



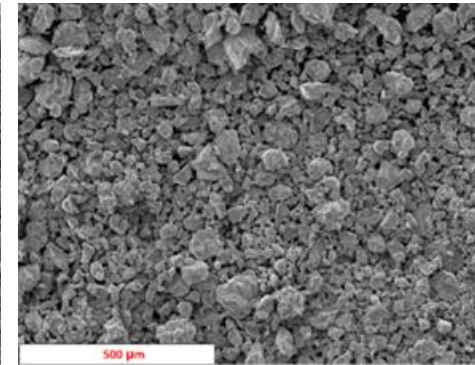
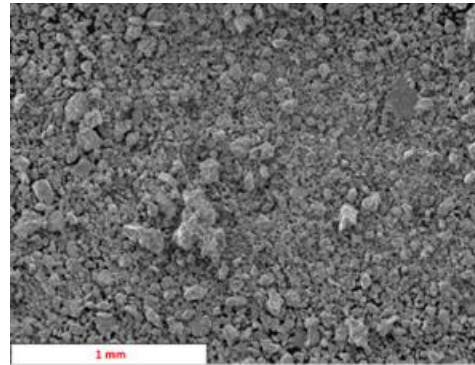
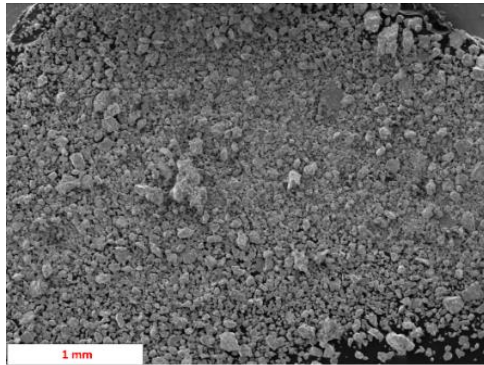
X50



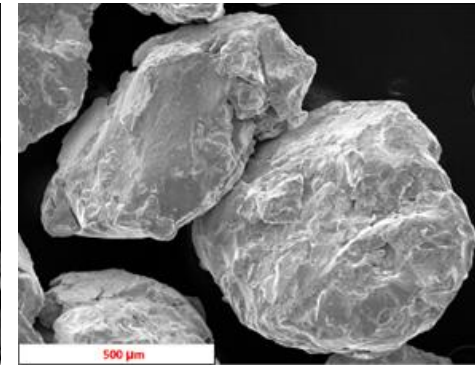
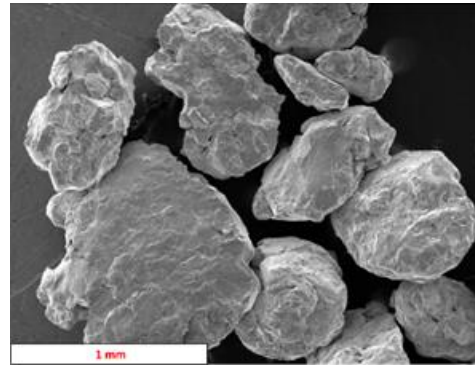
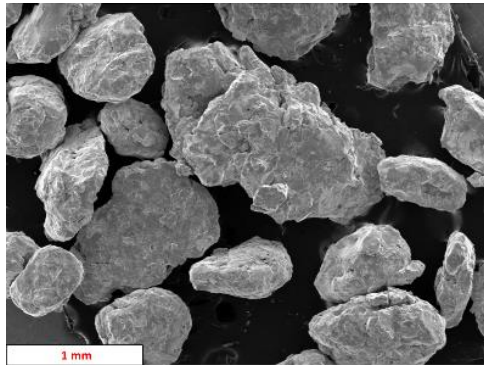
X100



BPR 4:1



BPR 8:1

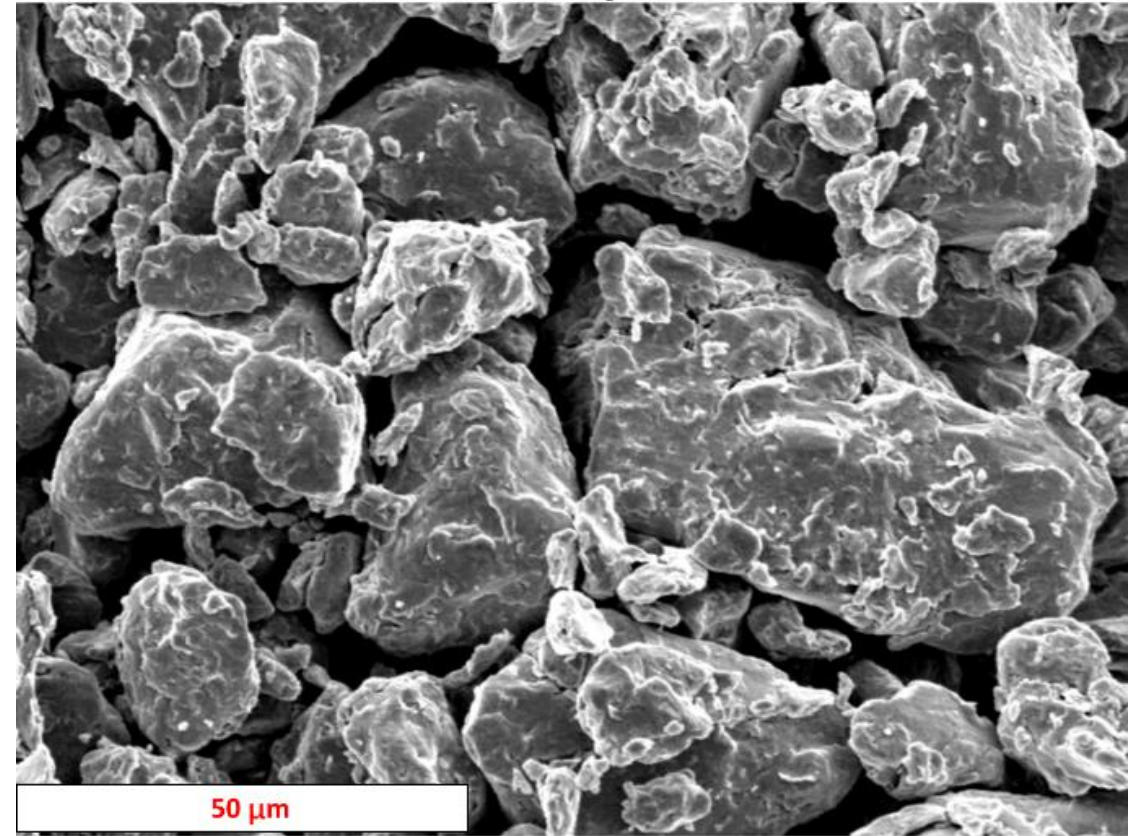
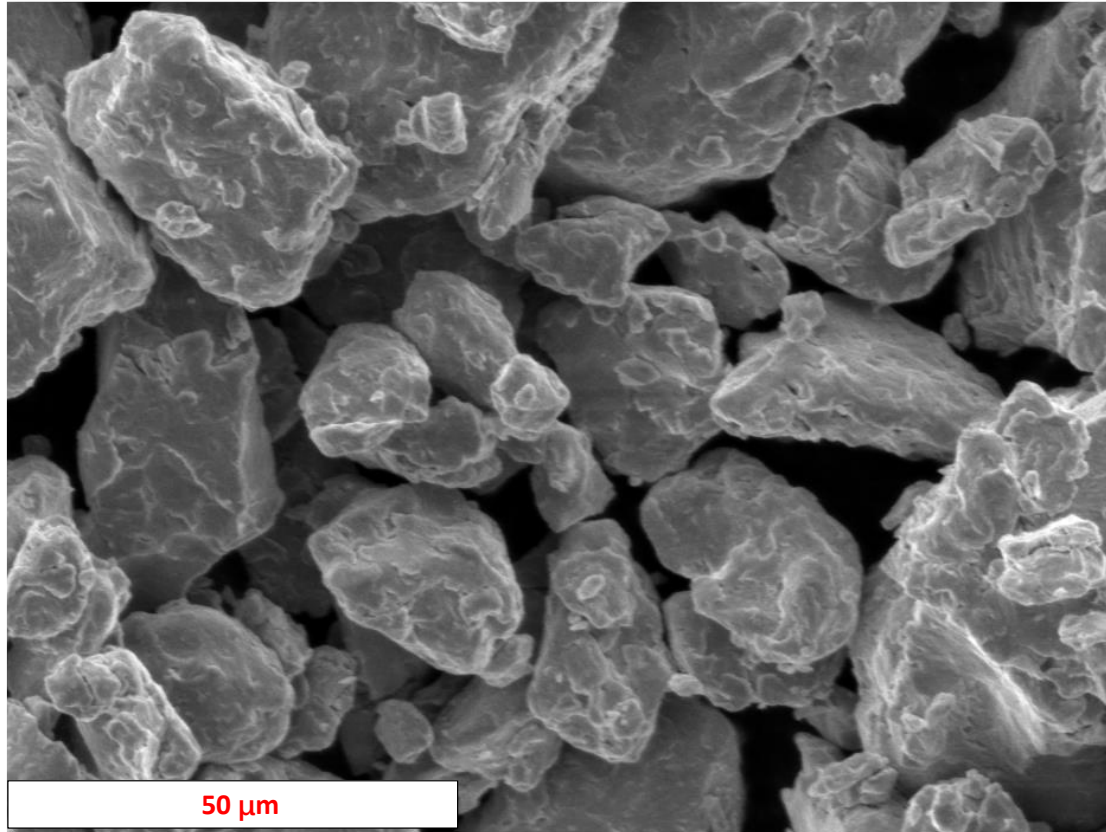


BPR 17:1

Ni_{71,25}Fe_{23,75}Al₅ 10 hours MA

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SEM



BPR 8:1

Ni_{71,25}Fe_{23,75}Al₅ and Ni_{71,25}Fe₁₉Mo_{4,75}Al₅

EDX

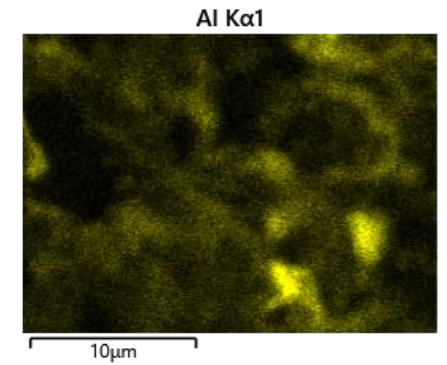
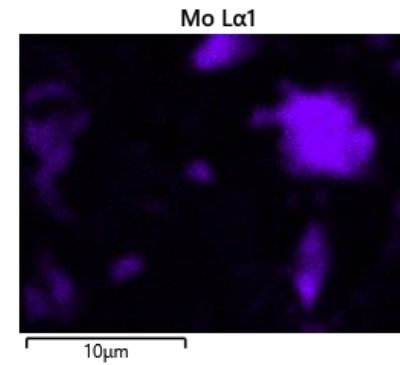
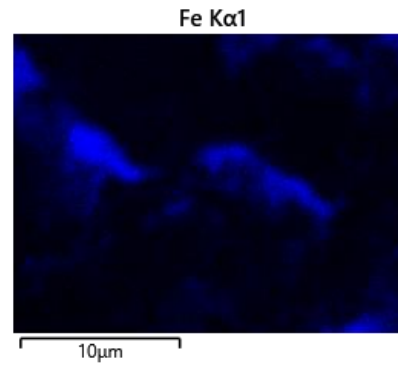
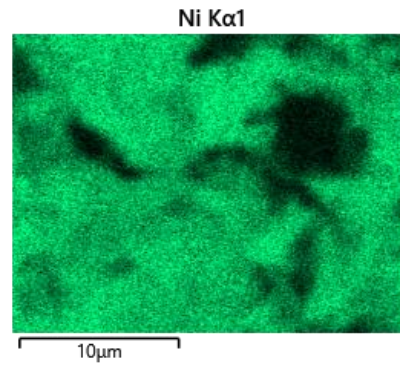
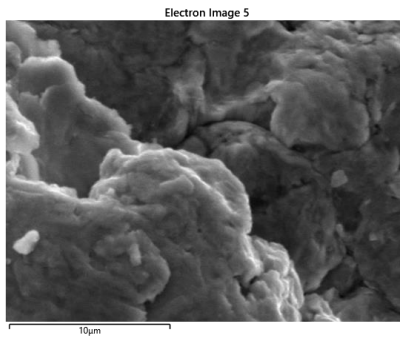
Ni

Fe

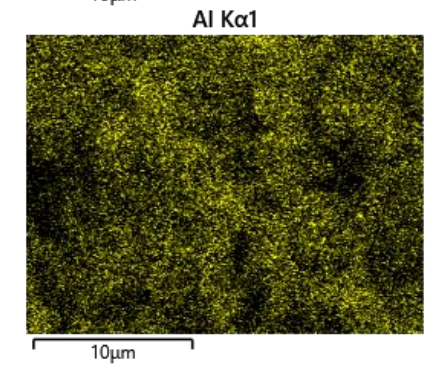
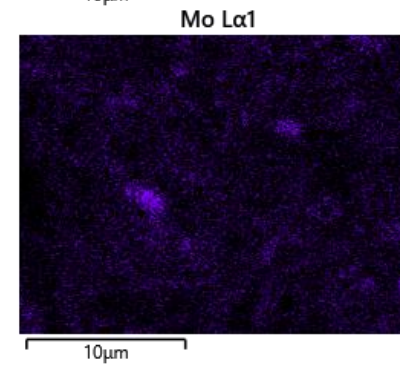
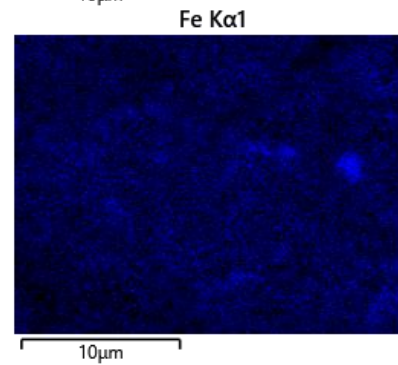
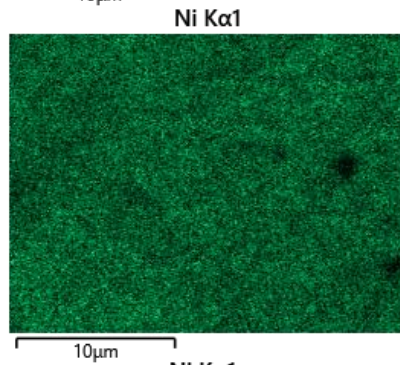
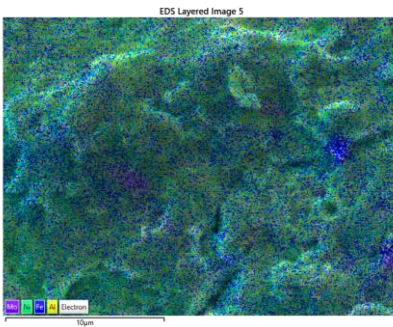
Mo

Al

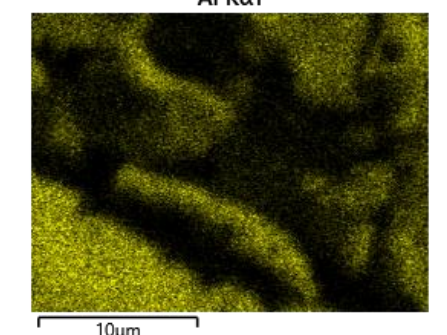
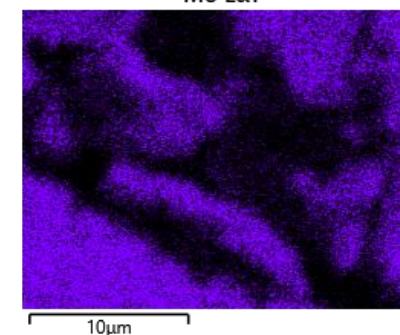
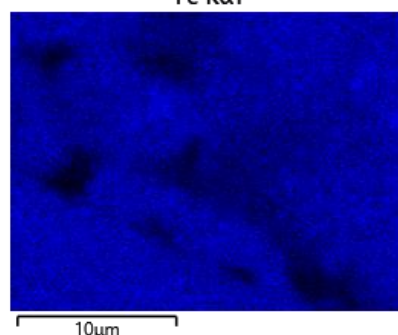
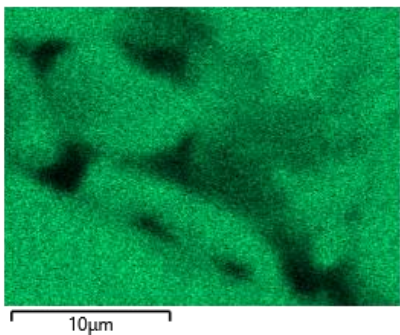
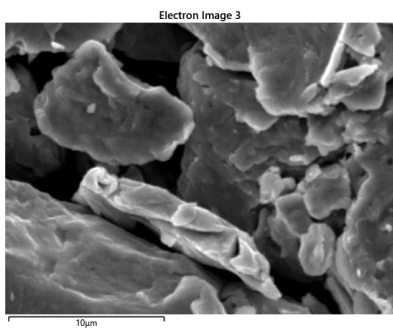
0.5 h MA



2 h MA



8 h MA



Ni_{70.5}Fe_{18.8}Mo_{4.7}Al₆

EDX

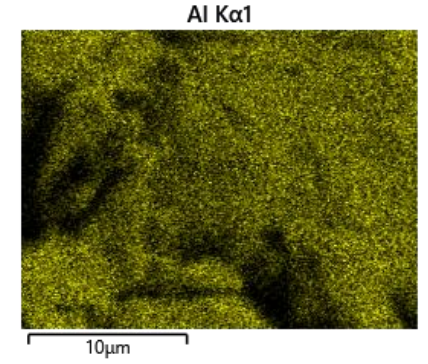
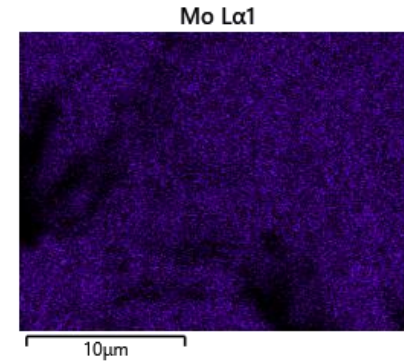
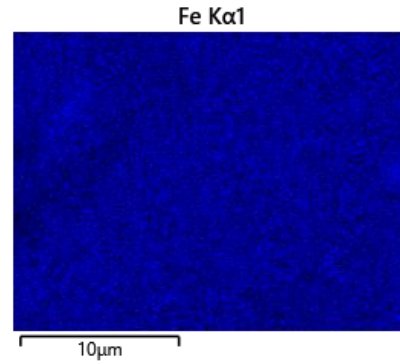
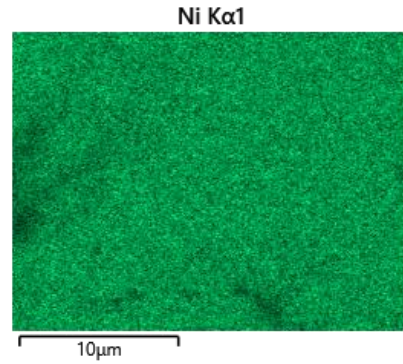
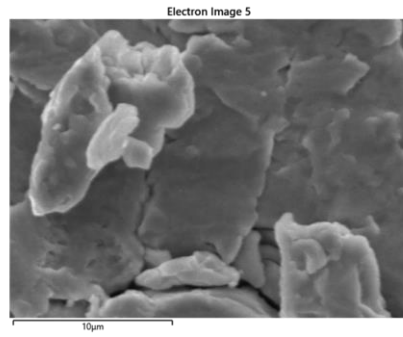
Ni

Fe

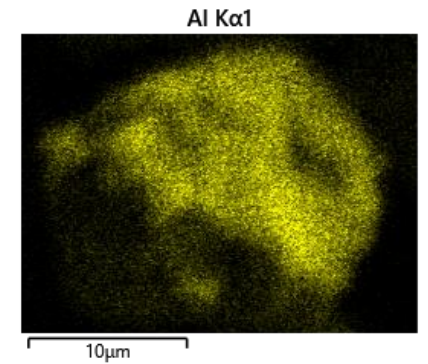
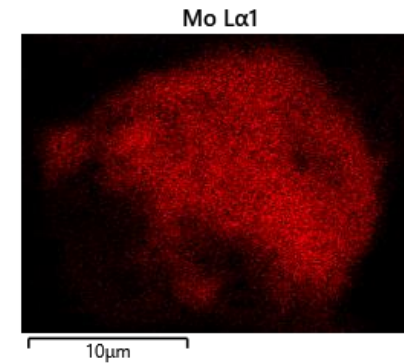
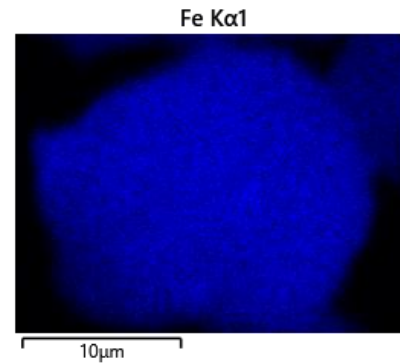
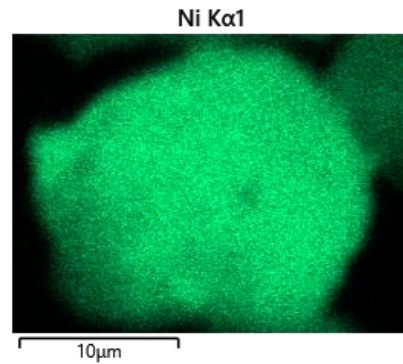
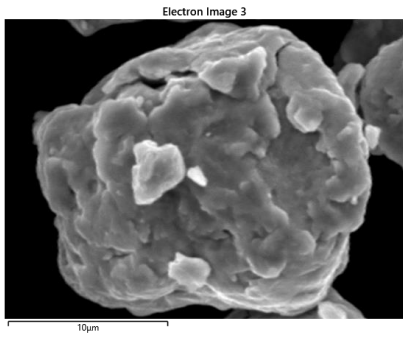
Mo

Al

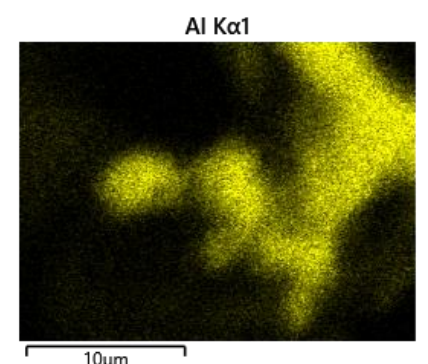
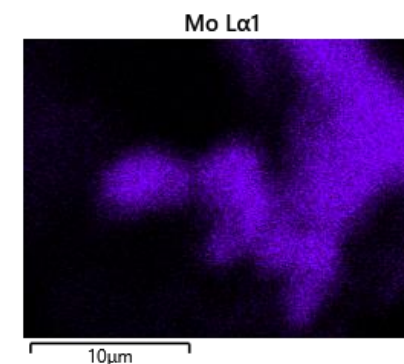
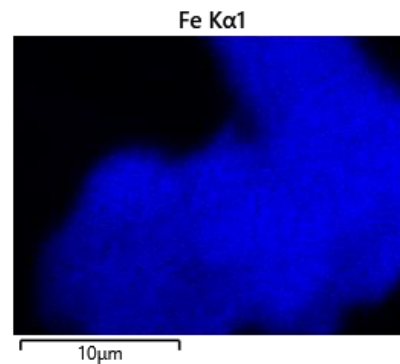
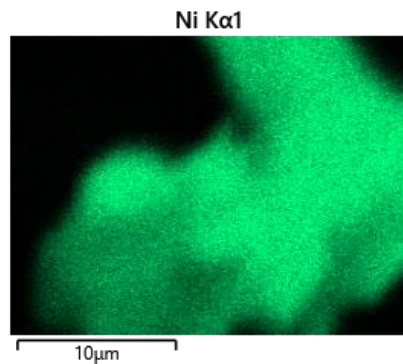
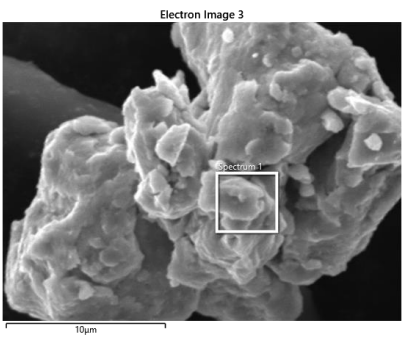
10 h MA



15 h MA



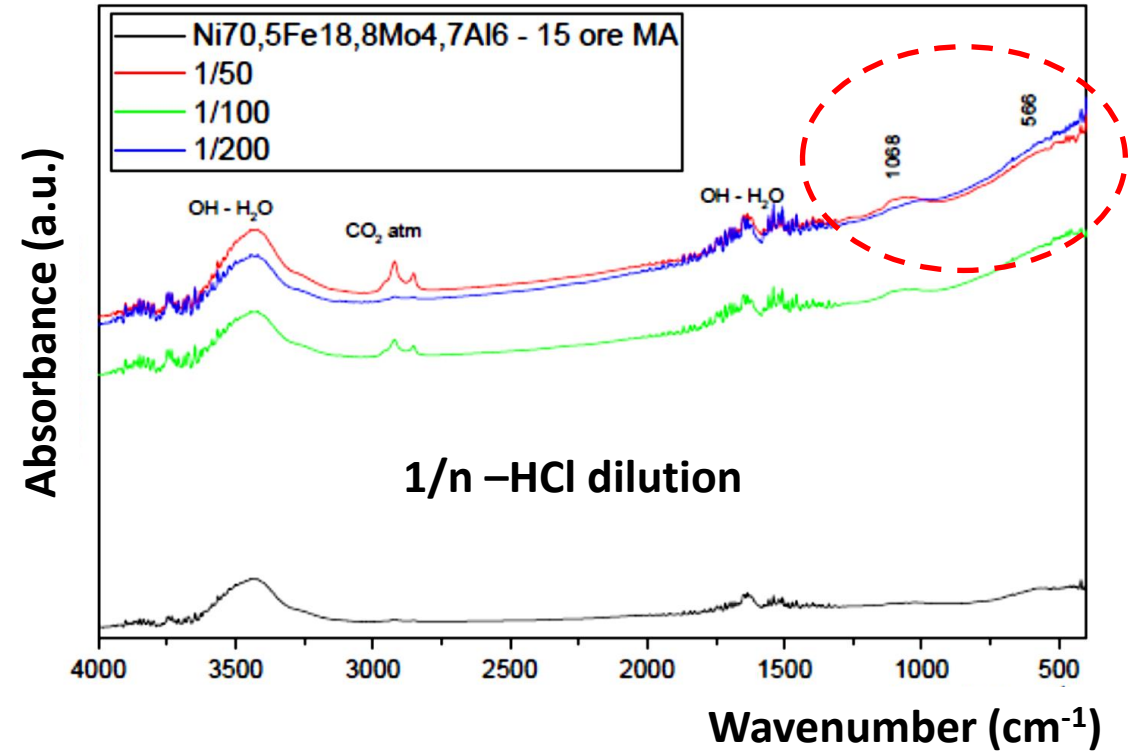
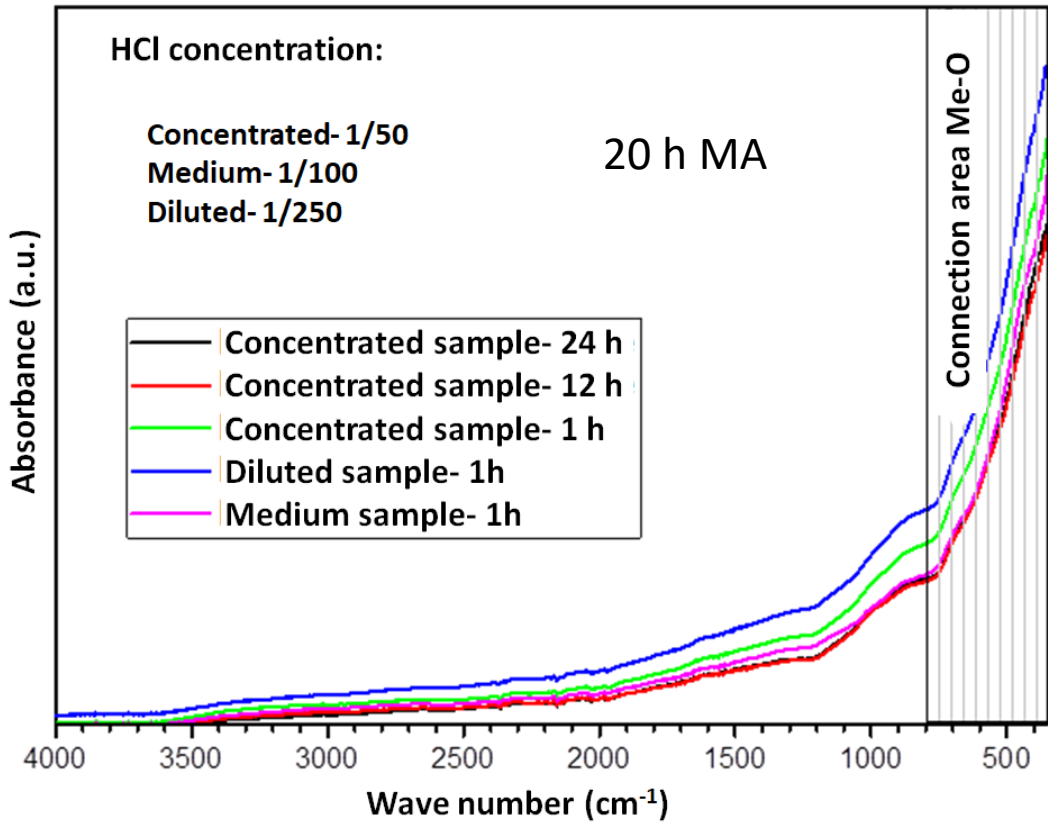
20 h MA



Ni_{70.5}Fe_{18.8}Mo_{4.7}Al₆

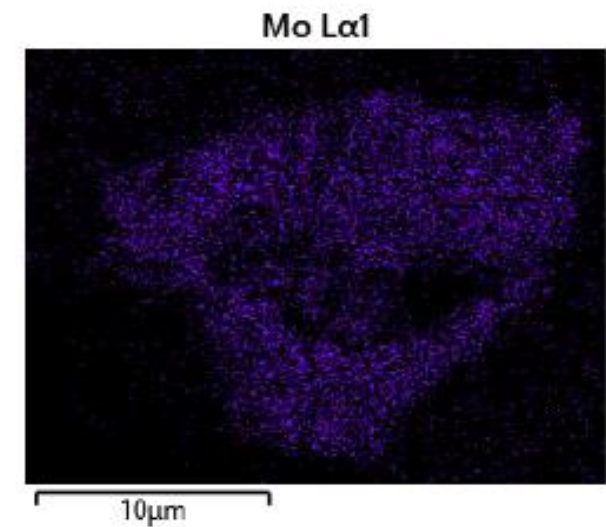
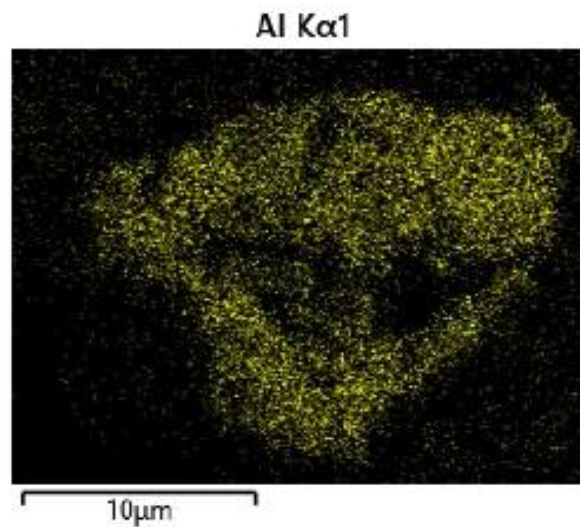
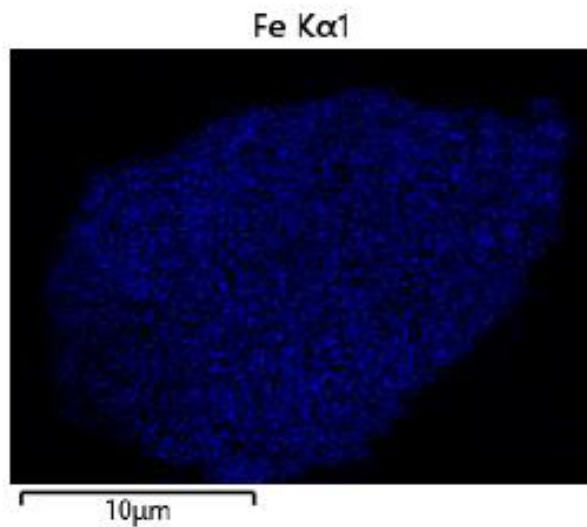
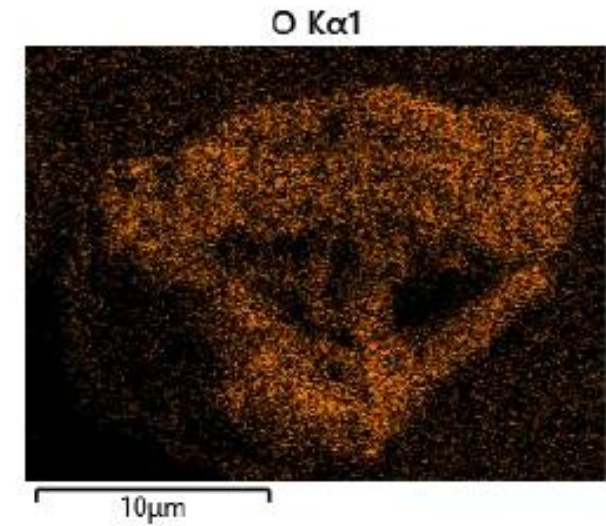
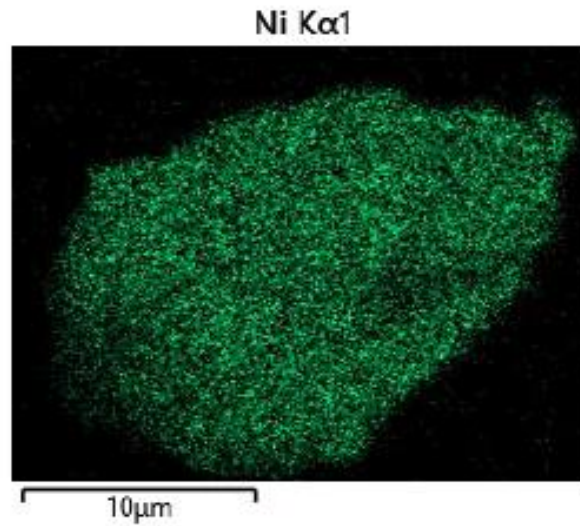
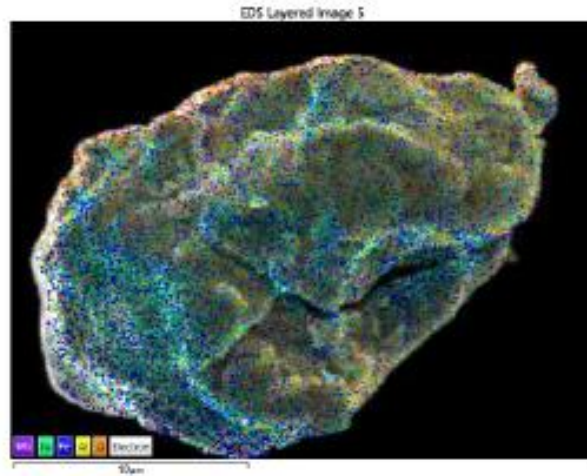
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FTIR-



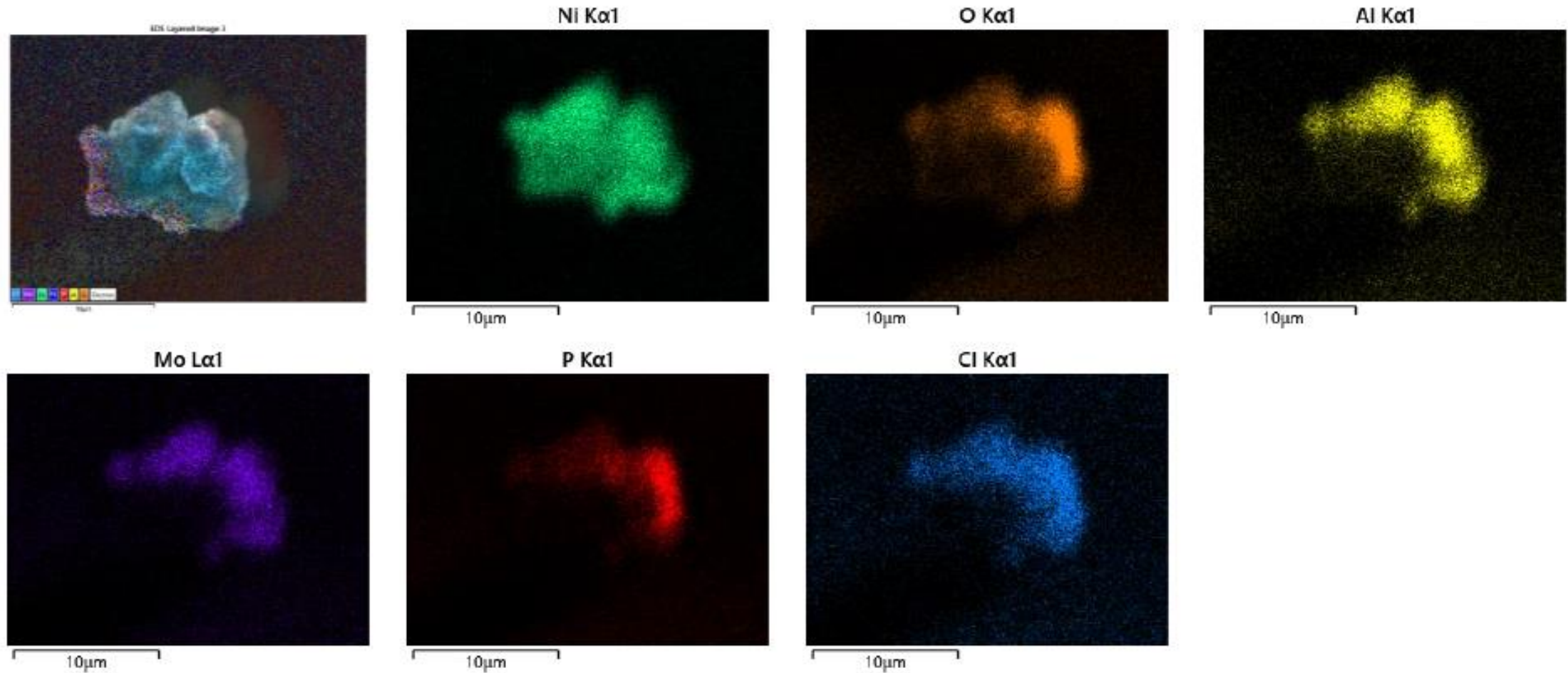
Ni71.25Fe19Mo4.75Al5 @oxide and Ni70.5Fe18.8Mo4.7Al6 @oxide (superficial oxidation in HCl)

SEM+EDX - Ni70.5Fe18.8Mo4.7Al6 @ oxide



superficial powder oxidations in air+Ar

SEM+EDX - Ni70.5Fe18.8Mo4.7Al6@oxide

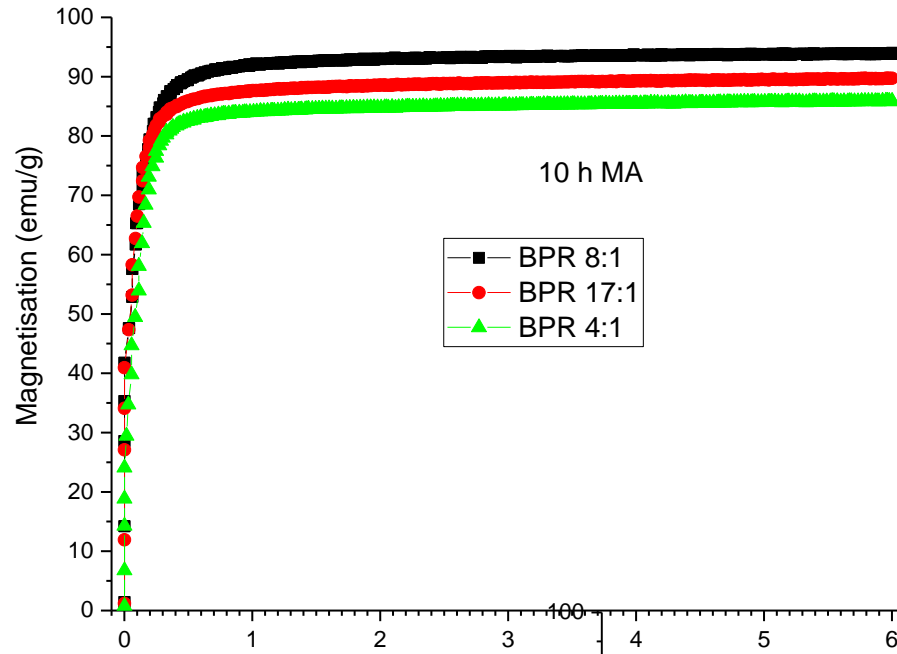
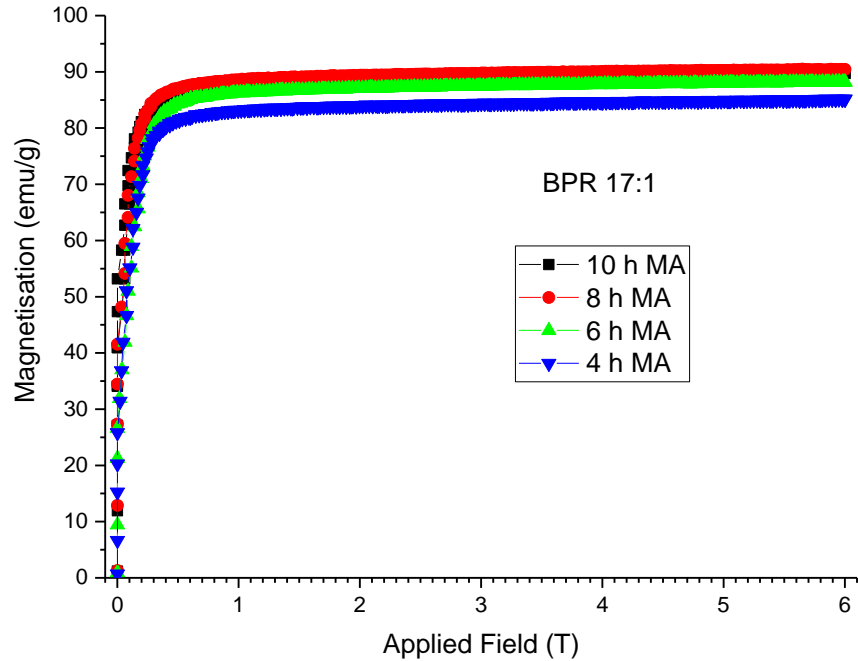


superficial powder oxidations by industrial burnishing kit

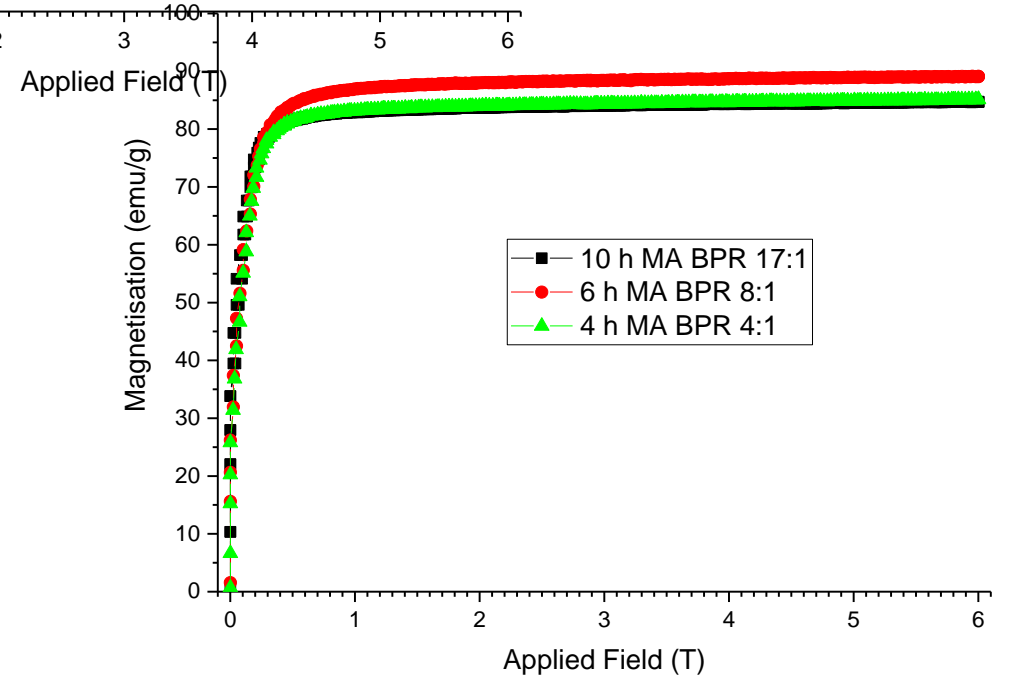
Magnetic characteristics

Magnetisation vs BPR

Magnetisation vs milling time



$\text{Ni}_{71,25}\text{Fe}_{23,75}\text{Al}_5$



Conclusions

- By mechanosynthesis up to 20 h and annealing **new alloys Ni70.5Fe18.8Mo4.7Al6, Ni71.25Fe19Mo4.75Al5 and Ni71.25Fe23.75Al5** as single phase was successfully obtained.
- The alloys are in nanocrystalline state, 15 nm after 15 h of mechanical milling.
- The particles size can be controlled by optimization of milling/alloying parameters
- The alloys were superficially oxidized in order to obtain **alloy@oxide** composite core-shell particles for sintering
- The addition of 5-6% wt. of Aluminium does not have a significant impact on magnetisation

Thank you for your attention!

Acknowledgement

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