PAVOL JOZEF ŠAFÁRIK UNIVERSITY IN KOŠICE
FACULTY OF SCIENCE

THE INFLUENCE OF TEMPERATURE ON ELECTRO-MAGNETIC PROPERTIES OF SOFT MAGNETIC COMPOSITES

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Košice 2023

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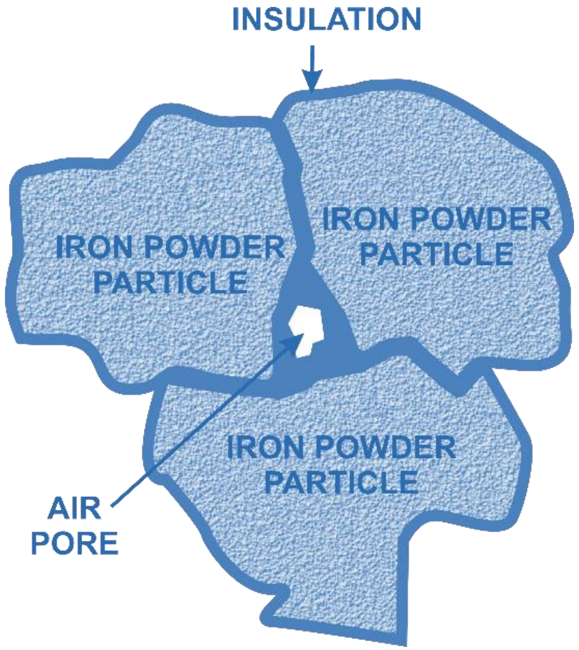
AIMS OF THE WORK:

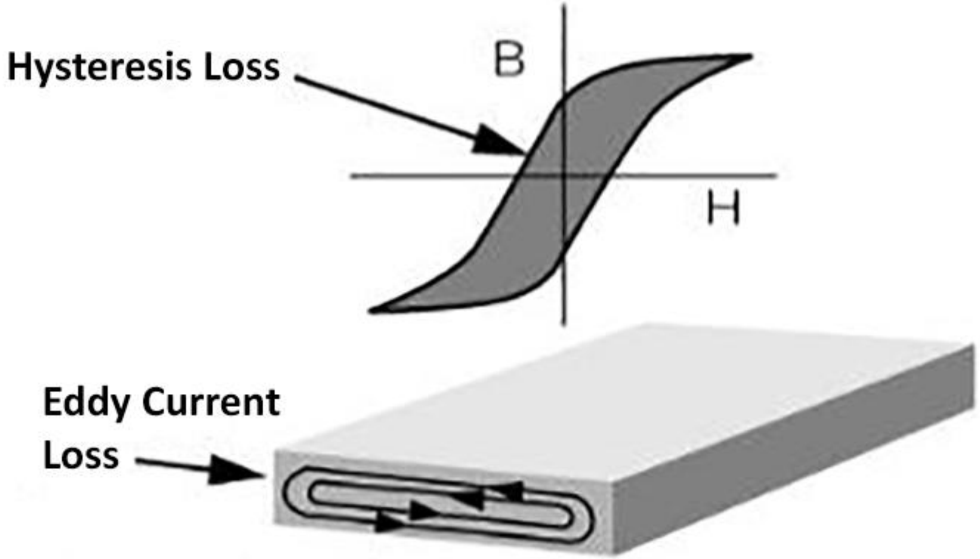
1. **Preparation of soft magnetic composites**

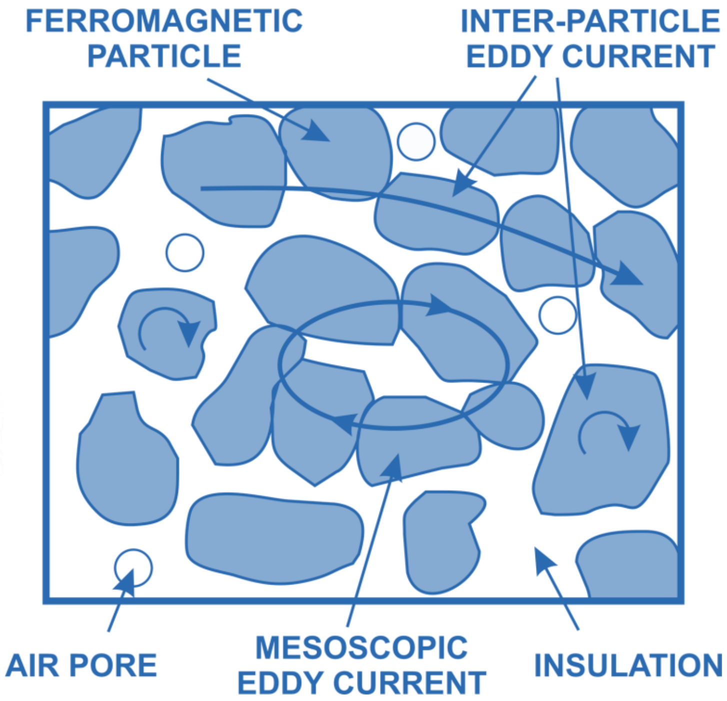
2. **Measurement of magnetic properties**

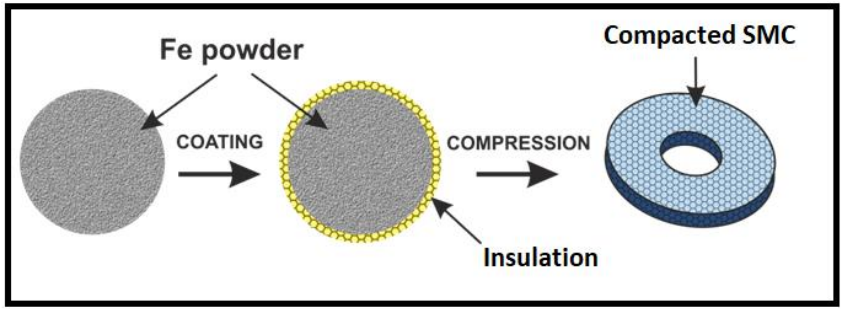
3. **Investigation of temperature dependence**

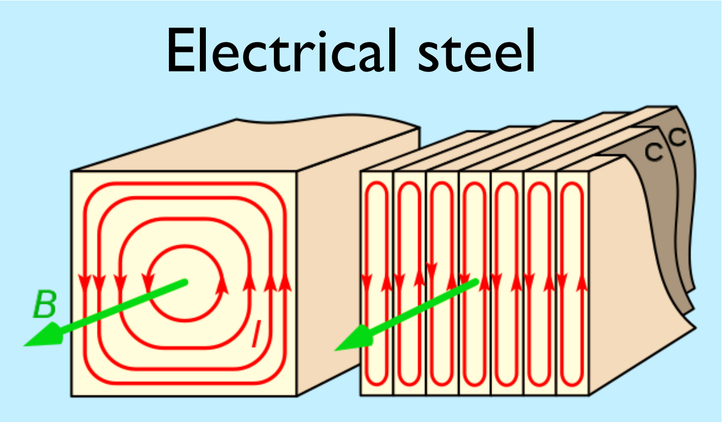
$$W(f) = W_{hyst} + W_{class}(f) + W_{exc}(f)$$

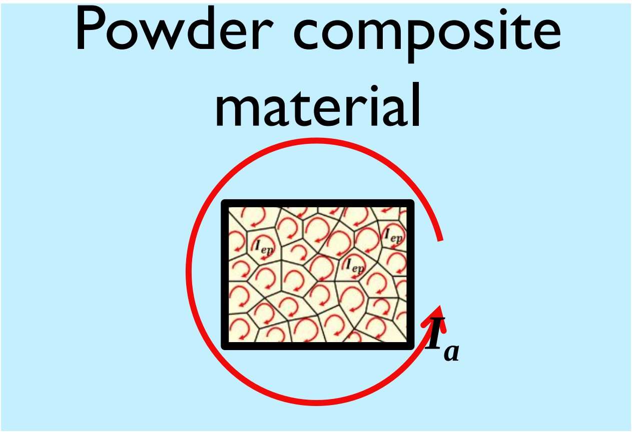










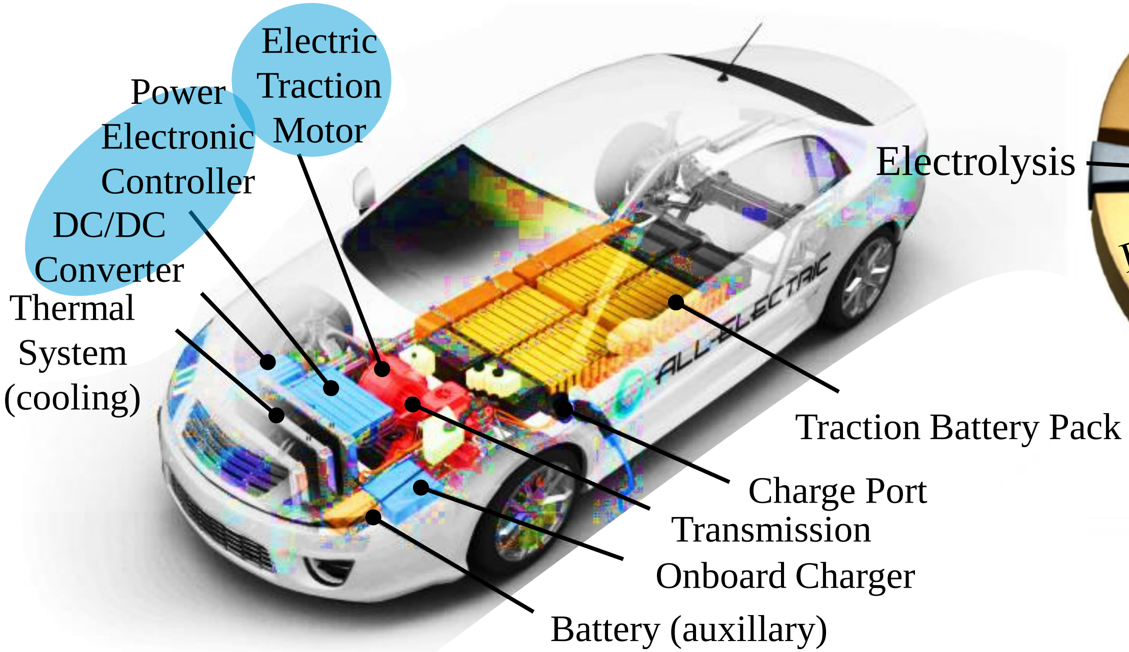


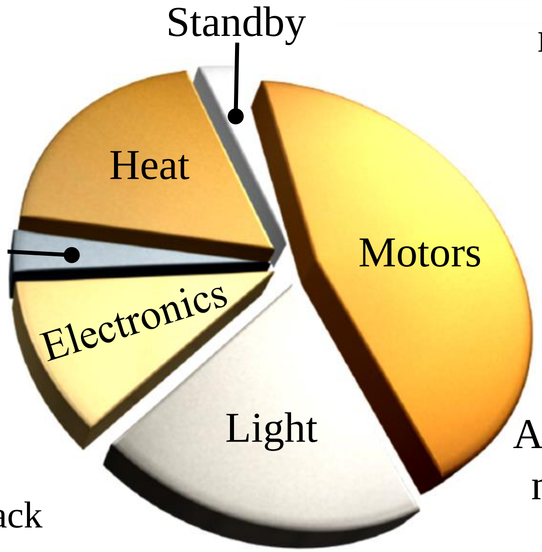
Common compacted SMC for magnetic measurements

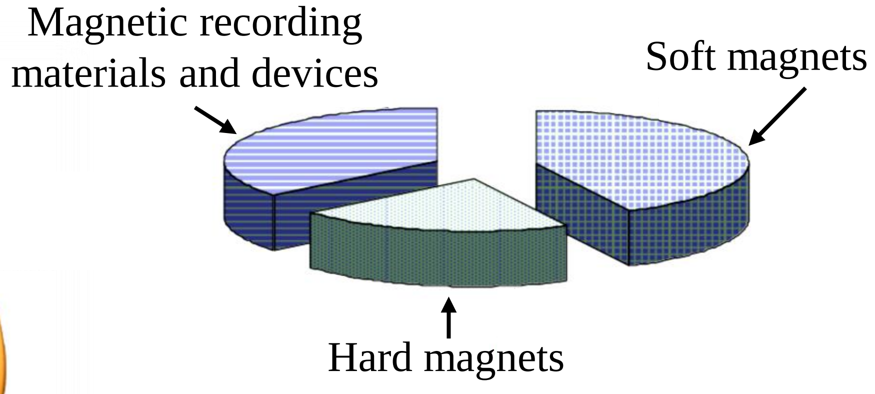
Reduced iron powder | **Insulated iron particle** | **Coated iron powder** | **Hydraulic press**

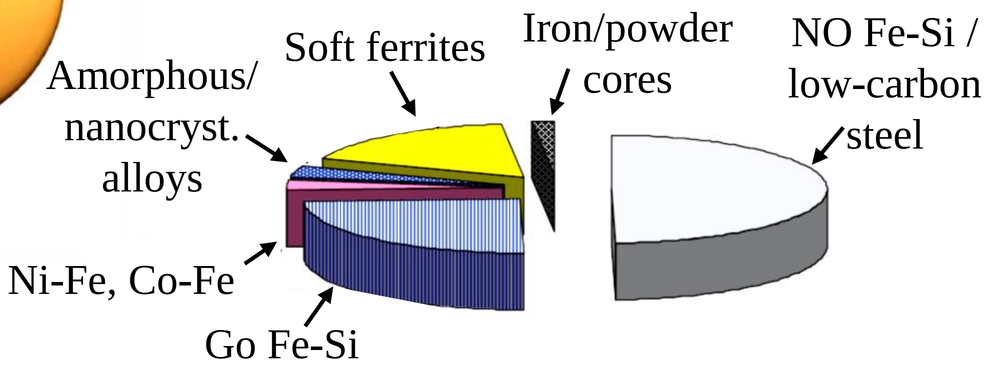
OR

ANY POSSIBLE SHAPE FOR WIDE SPECTRUM OF APLICATIONS LIKE:

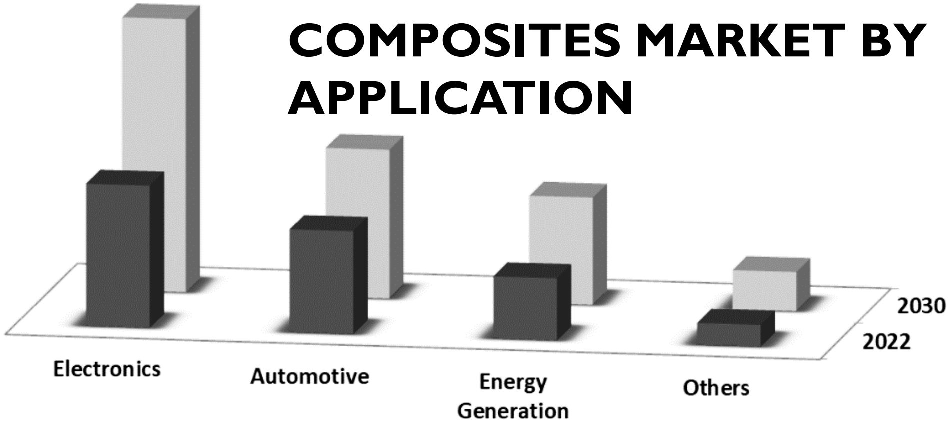


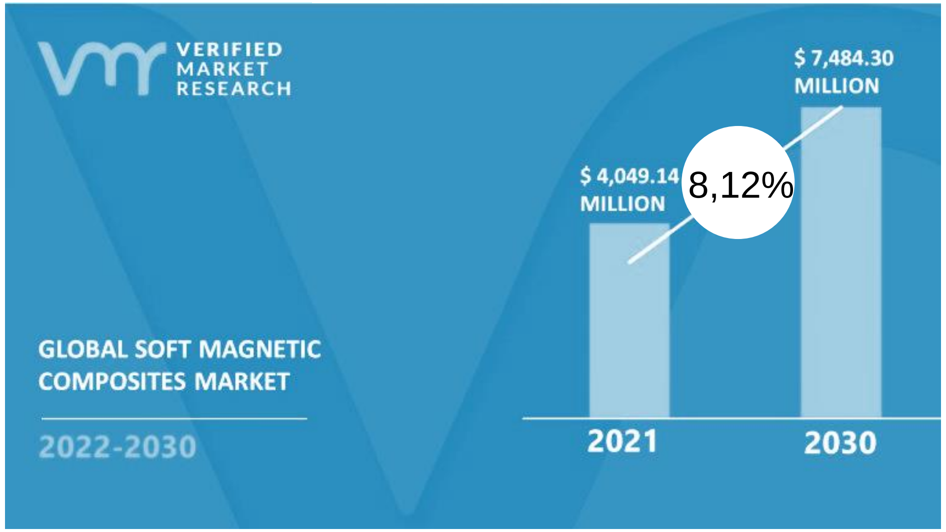






GLOBAL SOFT MAGNETIC COMPOSITES MARKET BY APPLICATION





SAMPLES PREPARATION:

Sintering

Particles milling (Retsch PM100)

Surface smoothing (Retsch PM100)

Dry-coating (LabRAM ResodynTM)

Wet-coating (Stöber method)

Powder metallurgy

Milling / smoothing

Planetary ball mill (Retsch PM100)

Laboratorium hydraulic press

Powder preparation

Matrix for preparation of ring samples

Stöber process

Resonant acoustic mixer (LabRAM ResodynTM)

SAMPLES CHARACTERIZATION:

- **Scanning electron microscopy (SEM)**
- **Energy-dispersive X-ray spectroscopy (EDX)**
- **Particle size distribution analysis (PSD)**
- **X-ray diffraction (XRD)**

JEOL JSM-7000F (SEM) with X-ray spectroscopy (EDX)

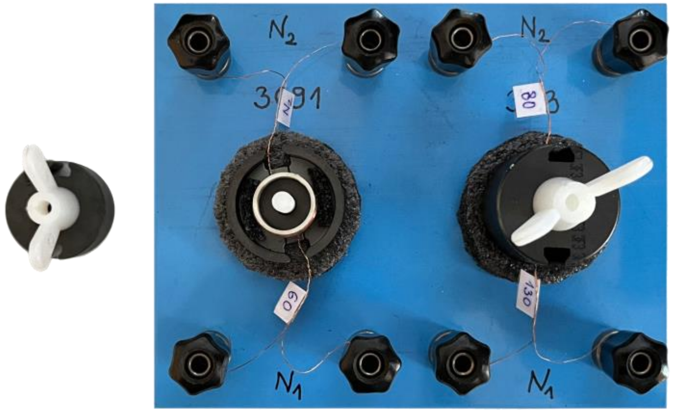
Laser scattering analyzer HORIBA LA-960 (PSD)

D8 DISCOVER X-ray Diffractometer (XRD)

MAGNETIC MEASUREMENTS:

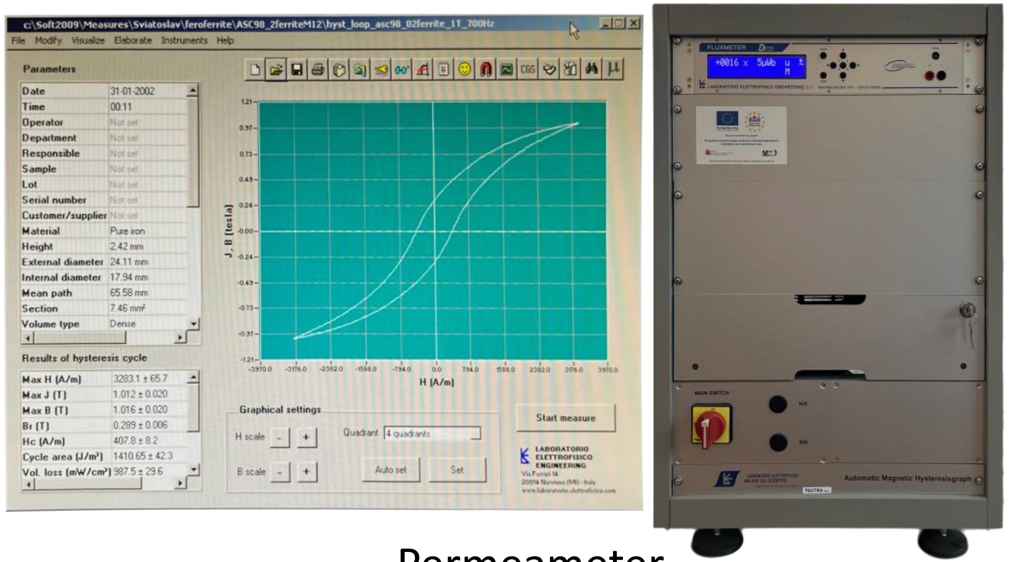
- **Electrical resistivity**
- **Normal magnetization**
- **Permeameter**
- **Complex permeability**
- **Energy losses**

Hysteresis-graphs
(MATS-2010M, MATS-2010SA)



Contactless method

LRC bridge
(HP 4194A)



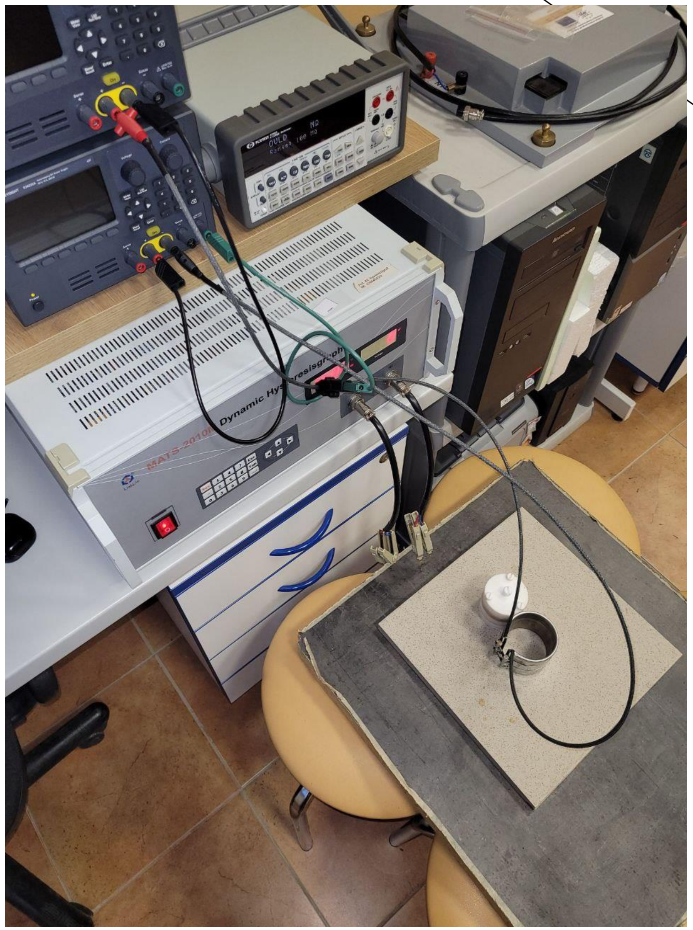
Permeameter
(AMH-1K-S Laboratorio Elettrofisico)

Sand heating

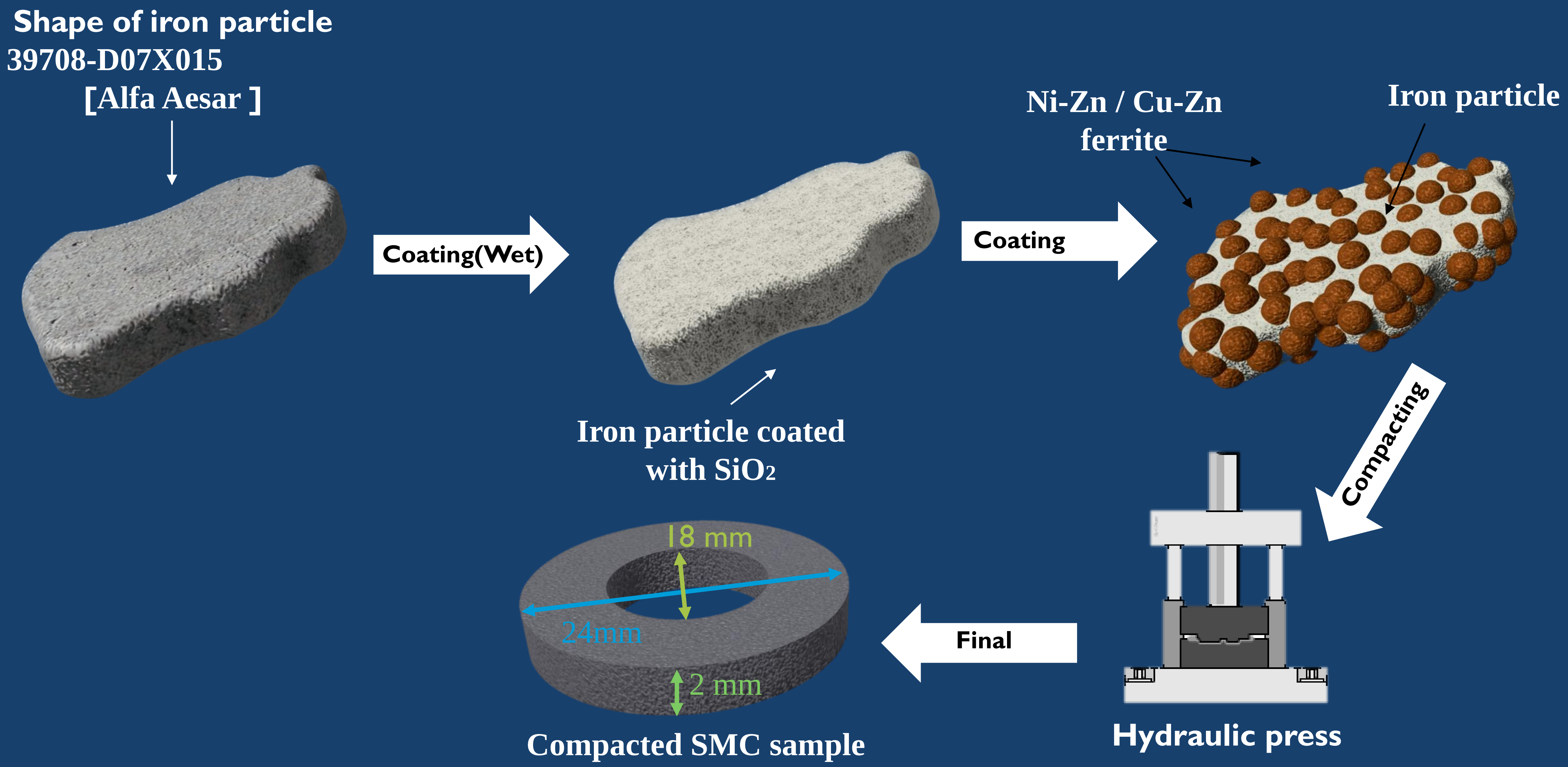
Teflon cell

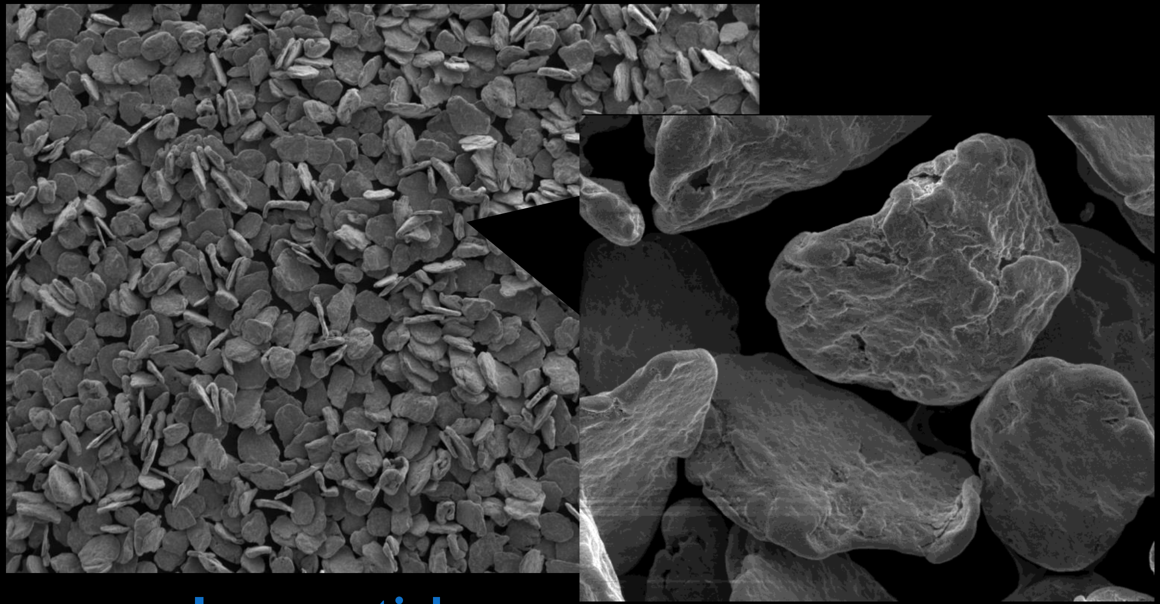
Resistive heater

Liquid cooling

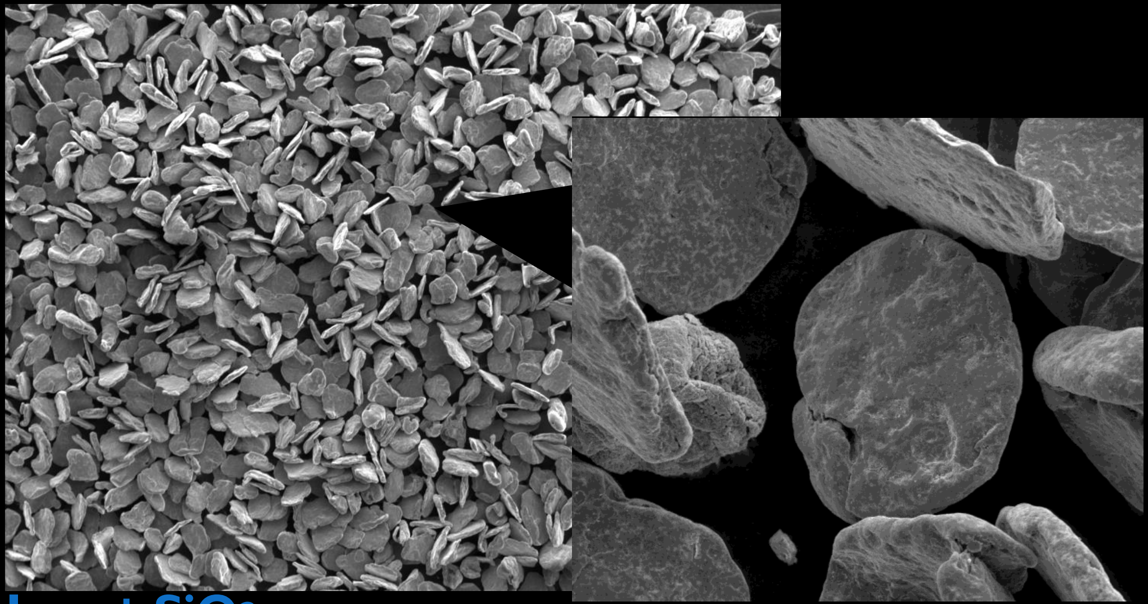


SMCs series №1 (Iron/SiO_2/ Ni-Zn/Cu-Zn ferrite)

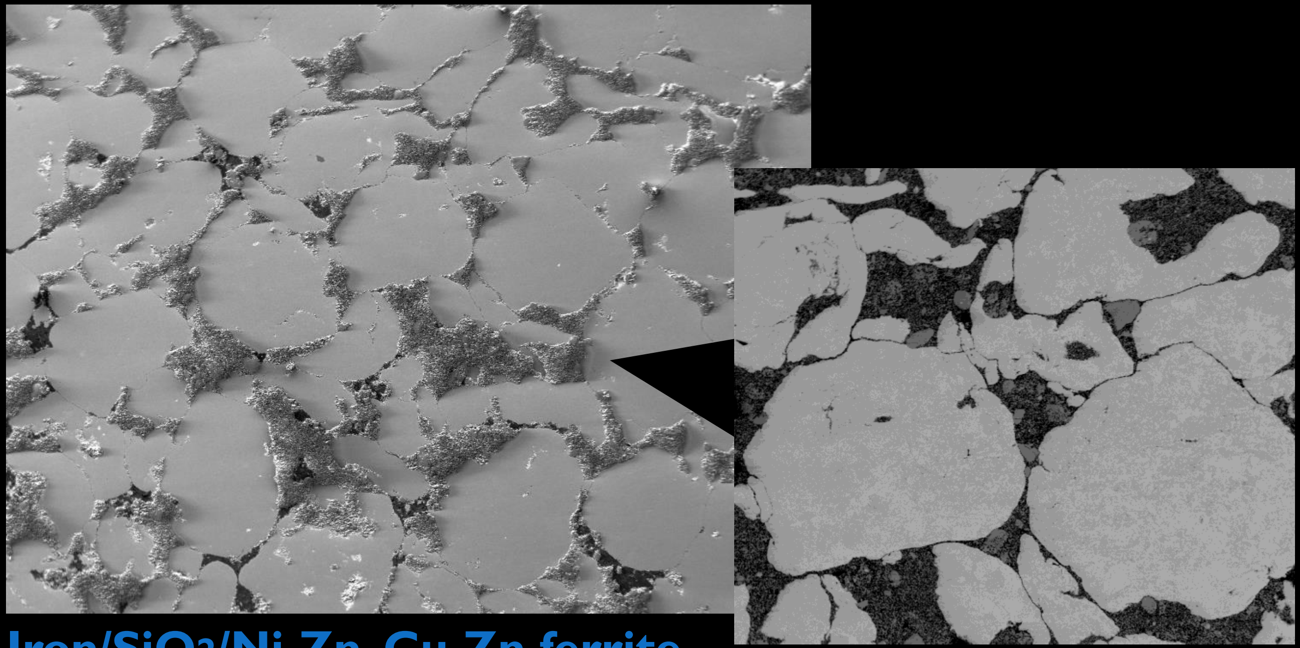




Iron particles (99.9% purity)

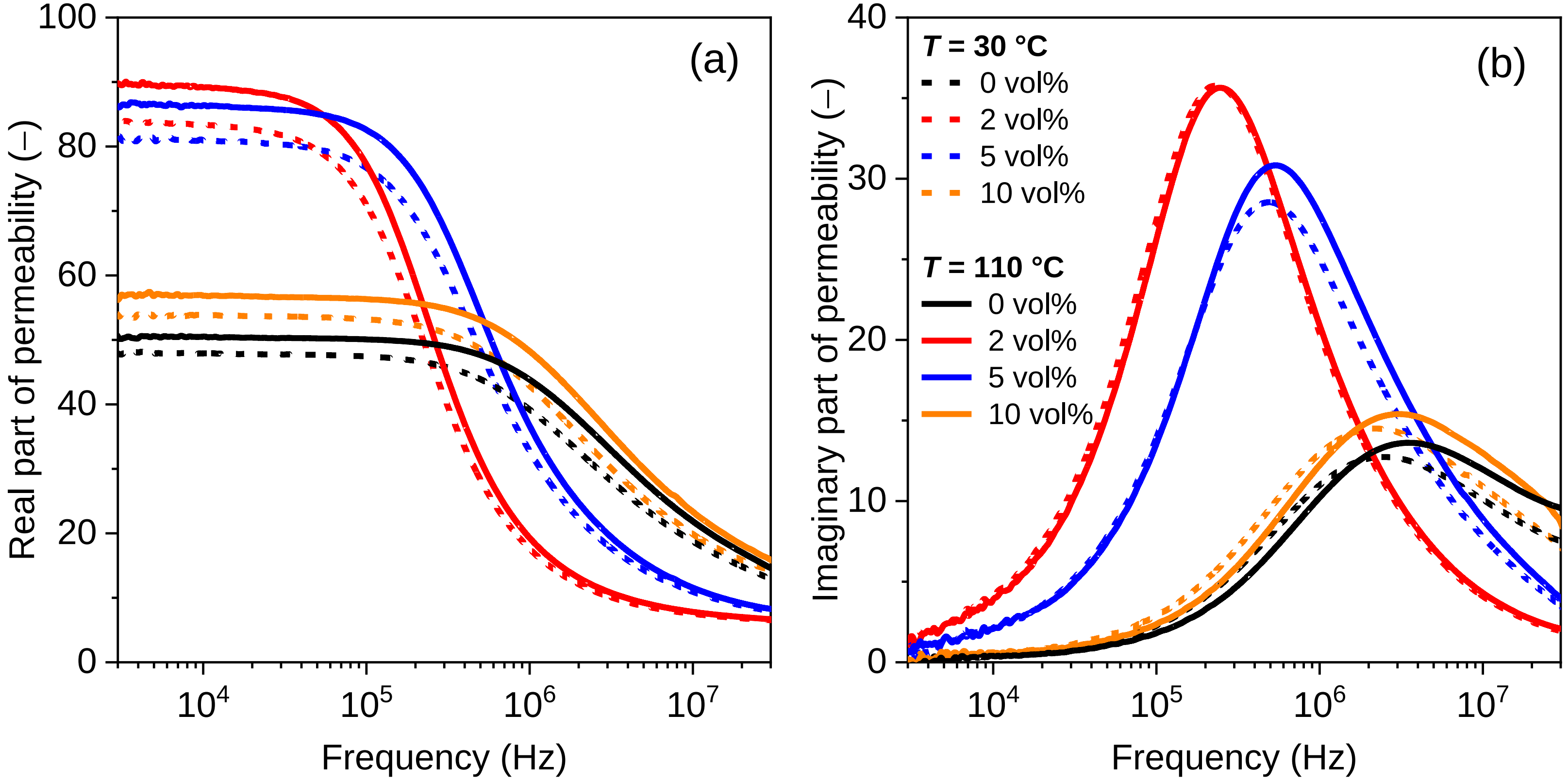


Iron + SiO_2



Iron/SiO_2/Ni-Zn, Cu-Zn ferrite

COMPLEX PERMEABILITY VERSUS TEMPERATURE



Complex permeability

$$\mu = \mu_{real} - j\mu_{imag}$$

Relaxation frequency

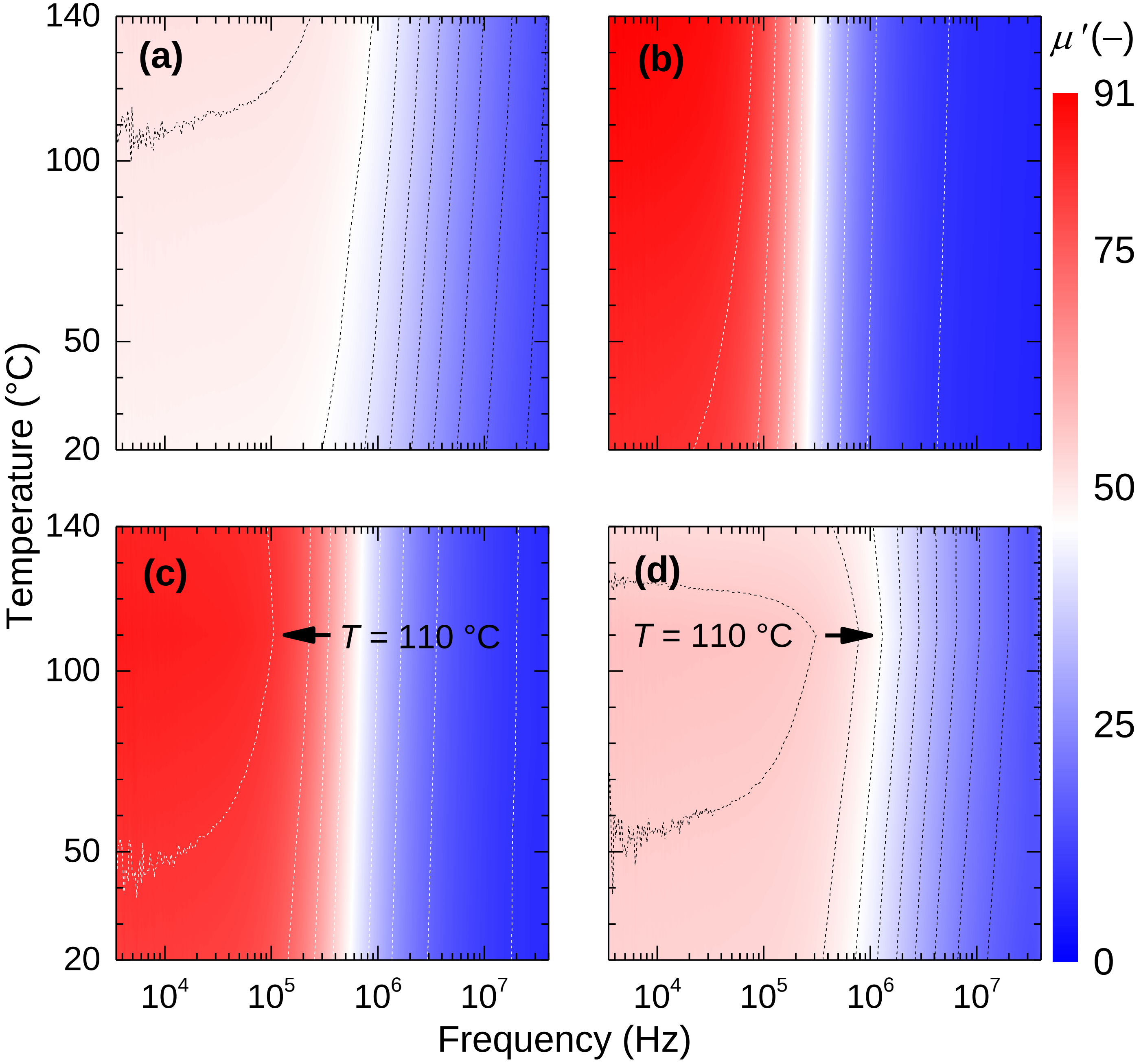
$$f_0 = \frac{4\rho}{\pi\mu_0\mu_{real}S}$$

Where ρ is electrical resistivity of the material, μ_0 is the permeability of free space and the S represents the cross-sectional area of the specimen.

(a) Real and (b) imaginary permeability of hybrid composites with different content of soft magnetic ferrite coating ranging from 0 to 10 vol% measured at temperature of 30 °C (dashed lines) and 110 °C (solid lines).

Sviatoslav Vovk, Samuel Dobák, Ján Füzer, Peter Kollár, Radovan Bureš, Mária Fáberová, Loss separation and thermal studies of Fe/SiO2/ferrite soft magnetic composites, *Journal of Alloys and Compounds* Volume 9455, 2023, Article number 169254.

Frequency–temperature maps of real component of permeability in composites with (a) 0 vol%, (b) 2 vol%, (c) 5 vol%, and (d) 10 vol% of ferrite. The maximum in permeability surface around 110 °C is marked by arrow in (c) and (d).

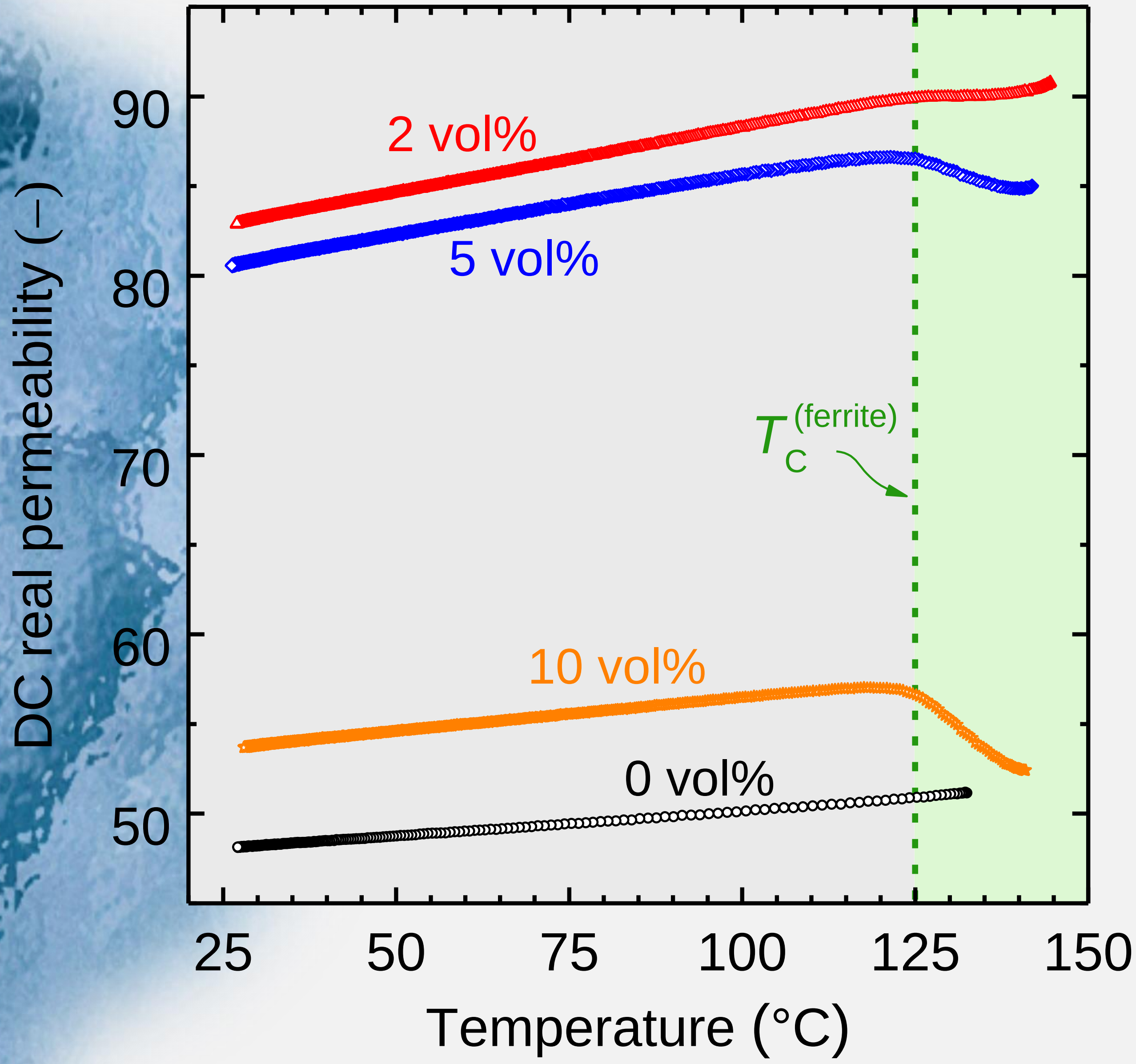


Resulting permeability

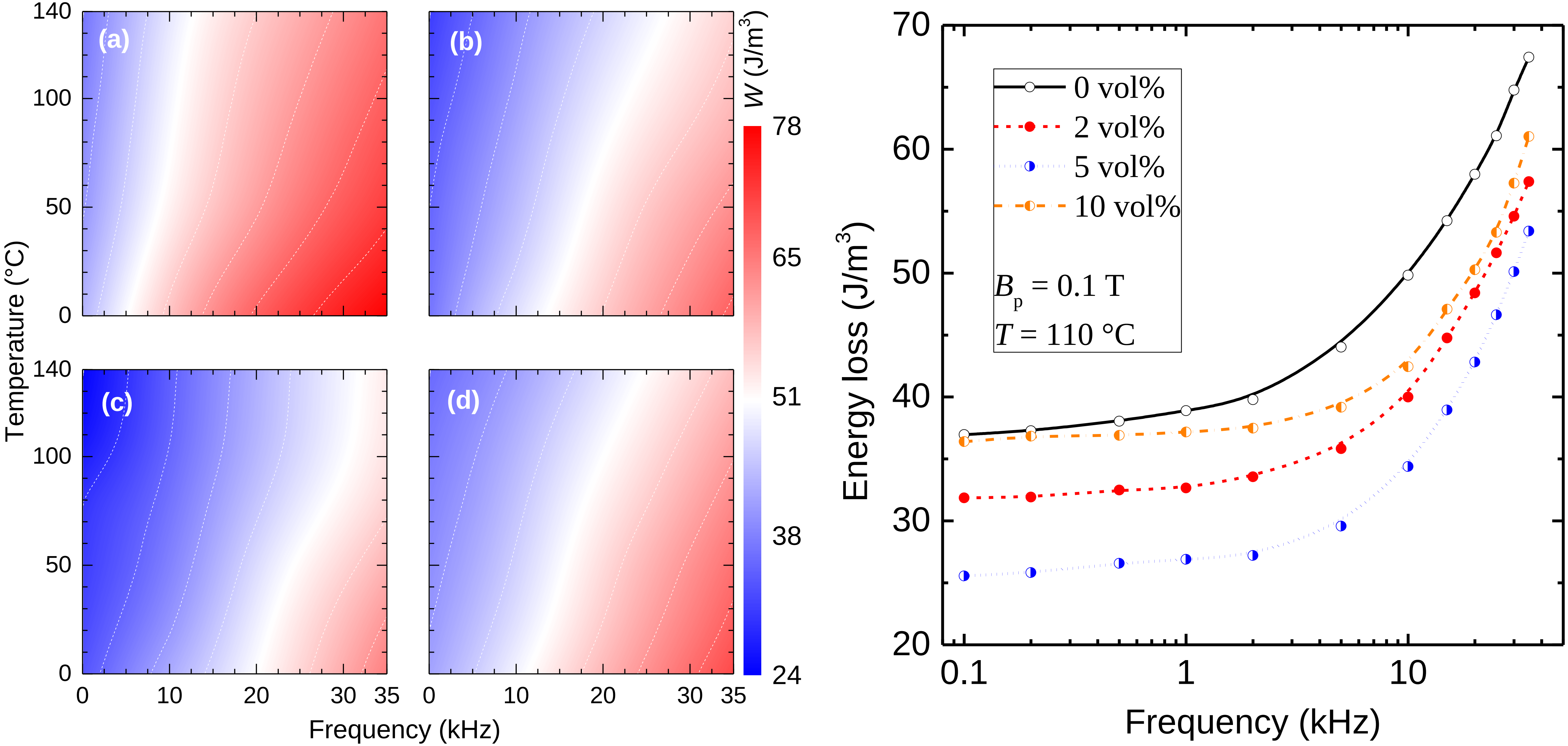
$$\mu \propto \frac{B_s^n}{|a\,K_1| + |b\,\sigma\,\lambda_s|}$$

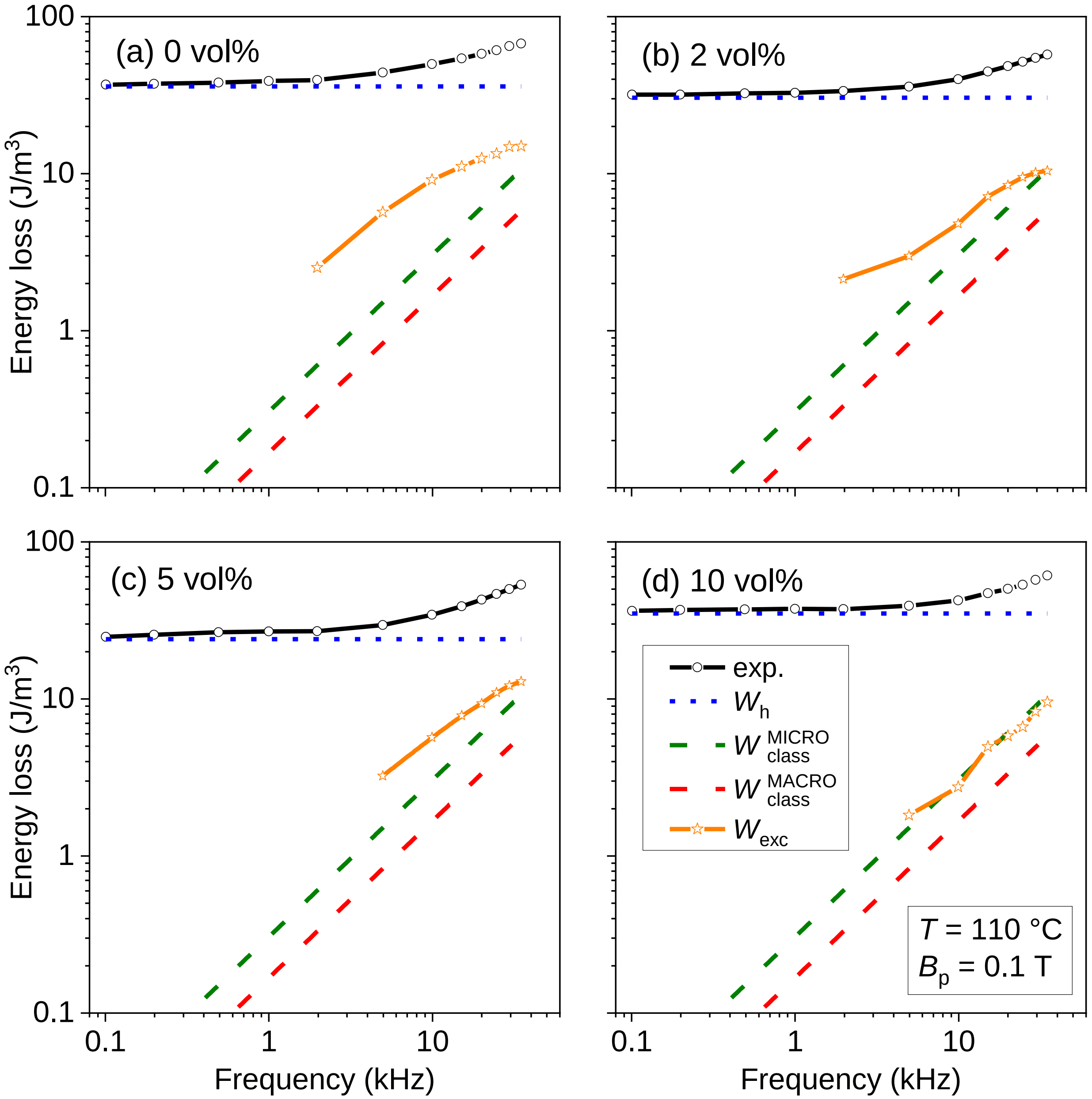
where B_S is the saturation induction, K_1 is the magnetocrystalline anisotropy constant, σ is the mechanical stress, λ_S is the saturation magnetostriction, and a, b, n are the material-dependent parameters

Temperature dependence of DC real component of permeability in hybrid composites with different content of ferrite.



ENERGY LOSSES VERSUS TEMPERATURE

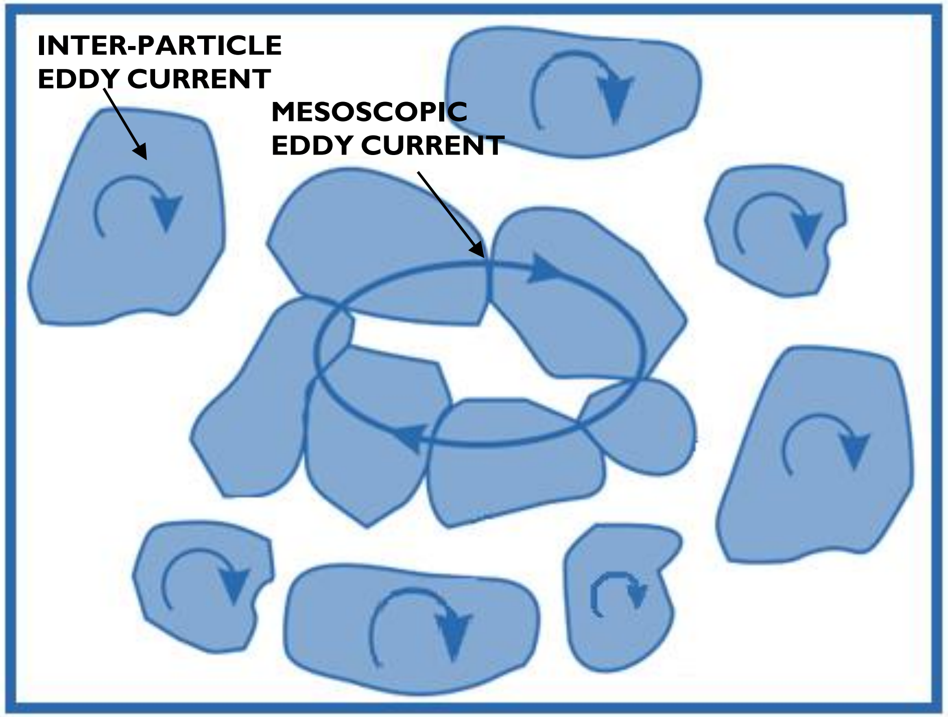




$$W(f) = W_h + W_{class}(f) + W_{exc}(f)$$

$$W_{class}(f) = W_{class}^{MICRO}(f) + W_{class}^{MACRO}(f)$$

$$W_{class}^{X}(f) = \frac{\pi^2}{6\rho}\, 12\, k(R)\, S\, B_p^2\, f$$



Conclusions: SMCs series №1

Ferrite content greatly affects Fe/SiO_2/ferrite composites' magnetic properties; a high Curie temperature ferrite is crucial for optimal performance.

Higher temperatures in iron/ferrite SMCs reduce loss increase at higher frequencies, with the lowest excess loss correlating to efficient domain wall movement.

Fe/SiO_2/2vol% ferrite composites exhibit an initial permeability of 84, stable across frequencies and uniform insulation coating with high resistivity yields an energy loss of 57 J/m^3 at 0.1 T and 35 kHz.

The use of ferrite as an insulating layer in the soft magnetic composites has tremendous potential in satisfying the stringent requirements within the high frequency range.

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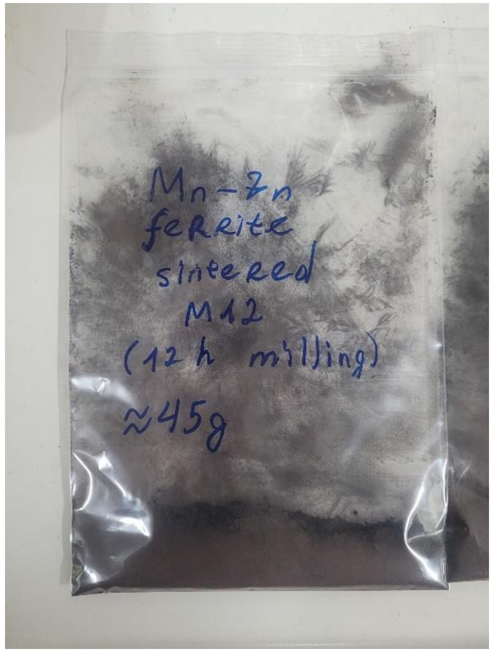
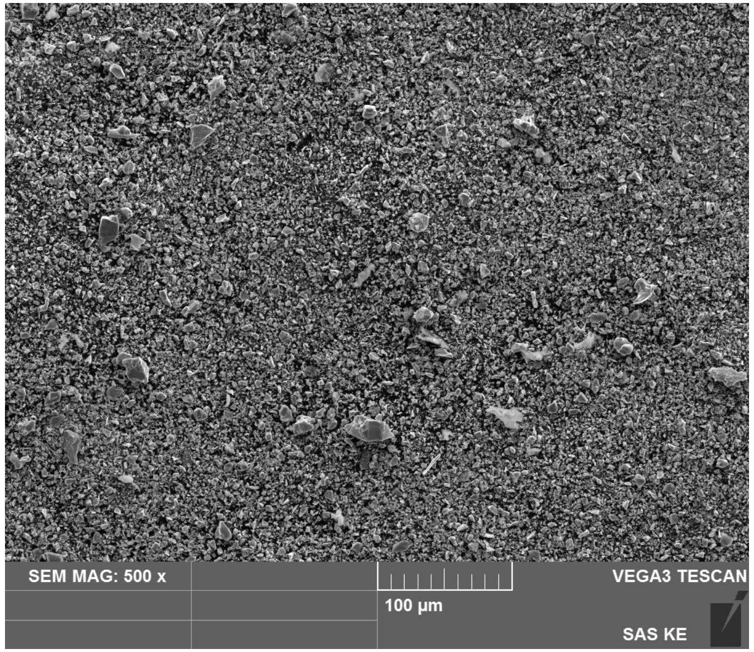
SMCs series №2 (Iron/Mn-Zn ferrite)

prof. Vassilios Zaspalis, PhD.

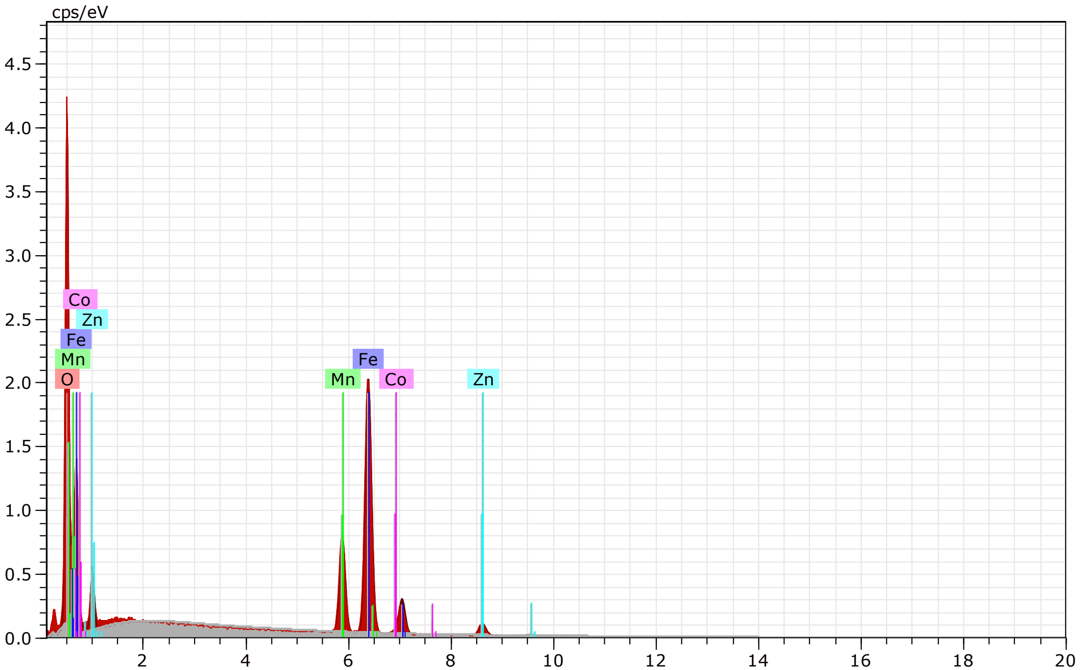
Vasiliki Tsakaloudi, PhD.

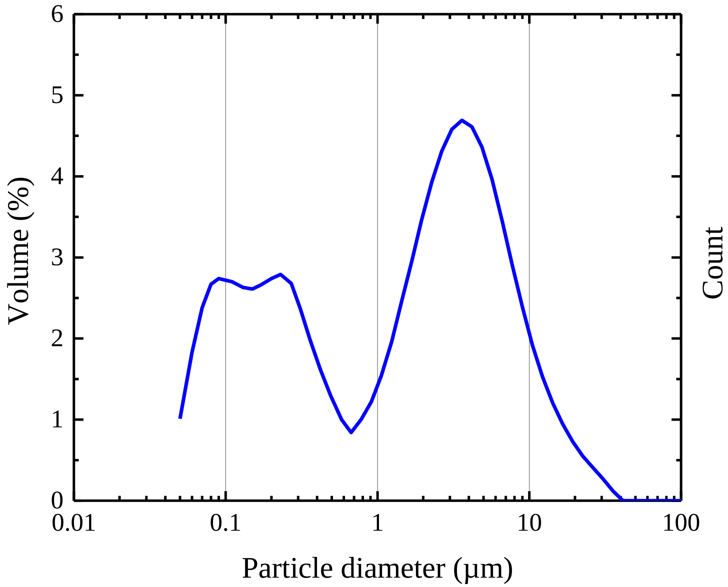


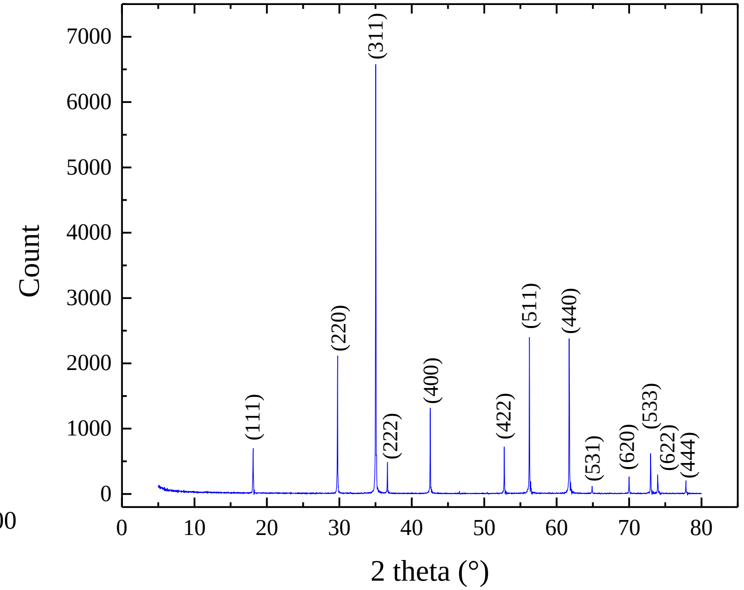


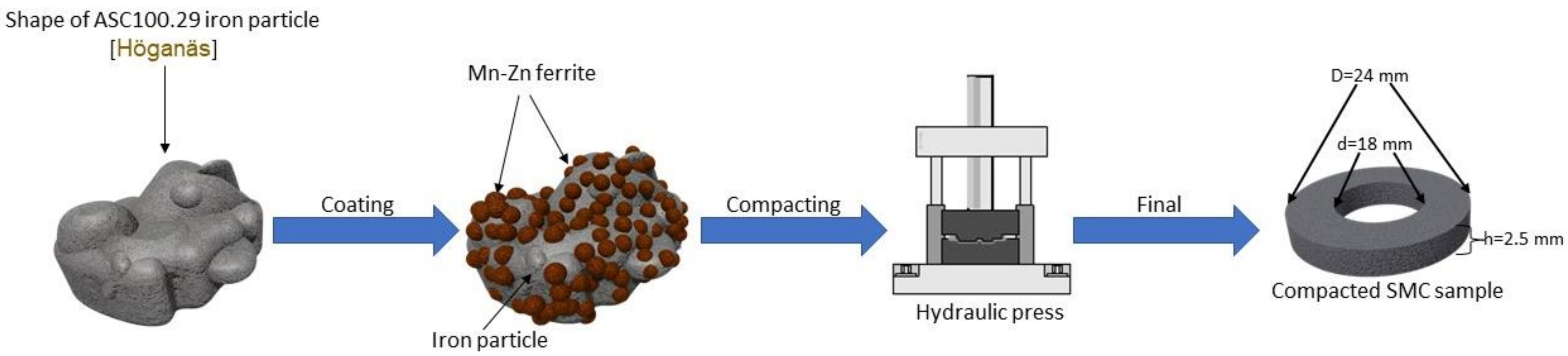
Spectrum: Spectrum

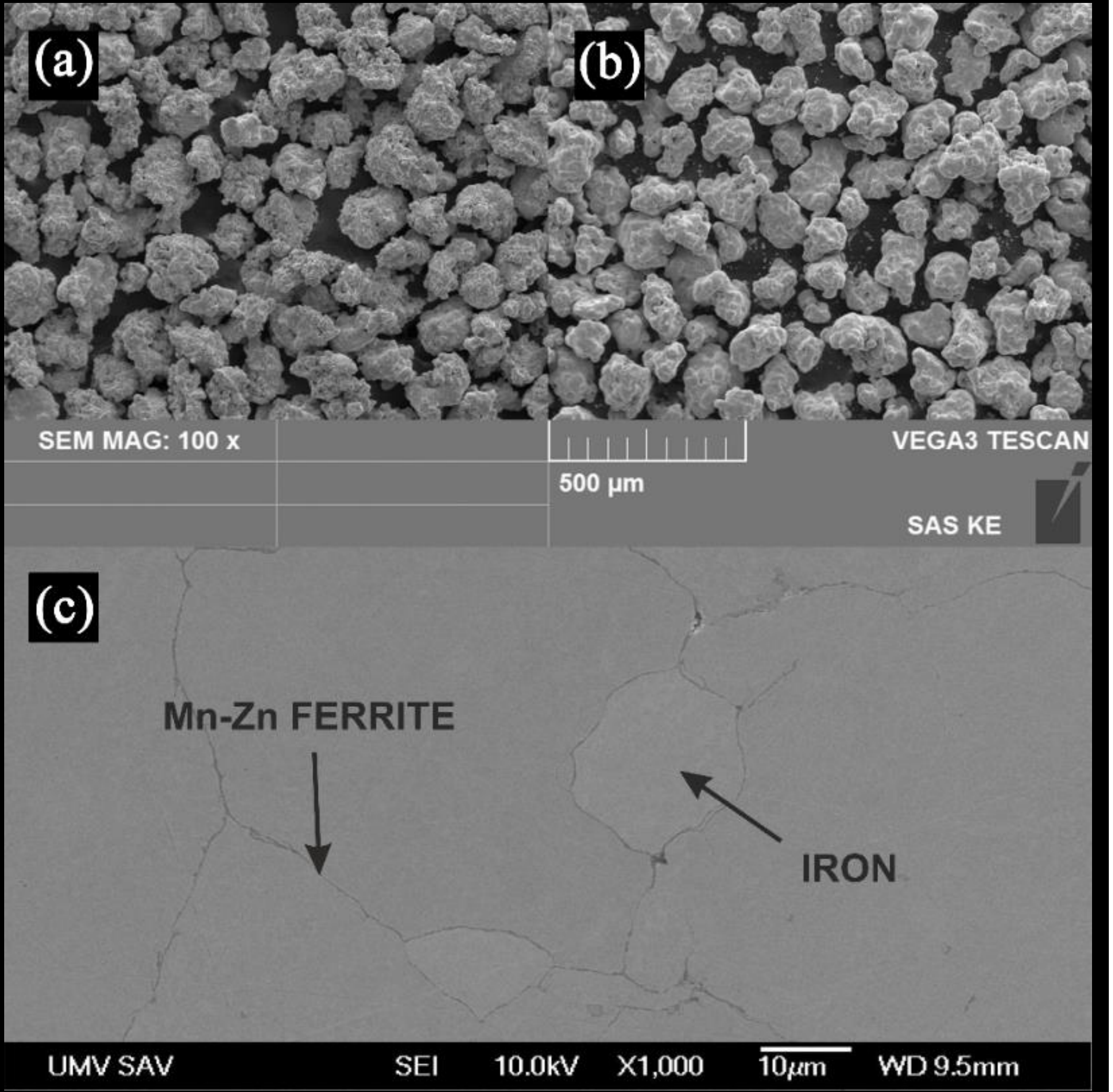
Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Oxygen	K-series	38,99	41,40	71,33	14,21
Manganese	K-series	11,51	12,22	6,13	1,05
Iron	K-series	38,61	40,99	20,23	3,21
Zinc	K-series	4,47	4,74	2,00	0,57
Cobalt	K-series	0,60	0,64	0,30	0,18
	Total:	94,18	100,00	100,00	

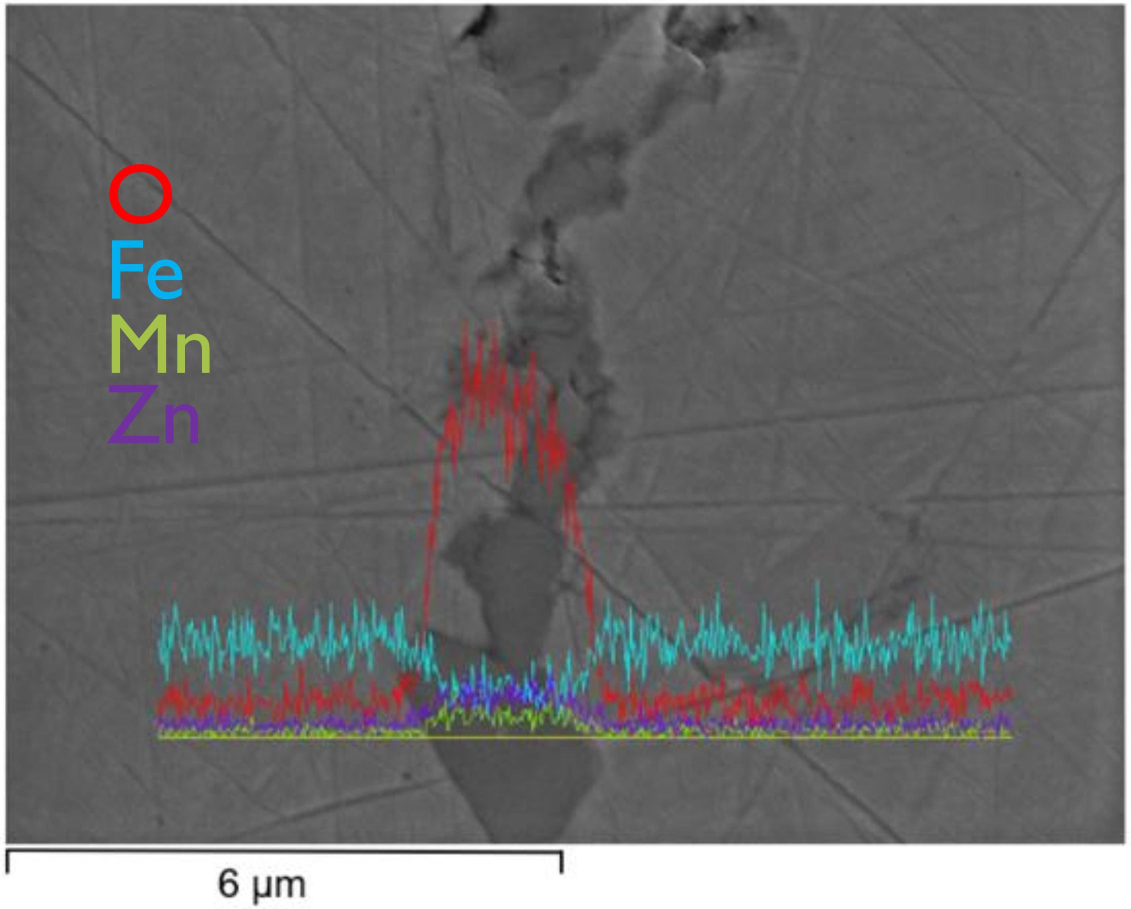


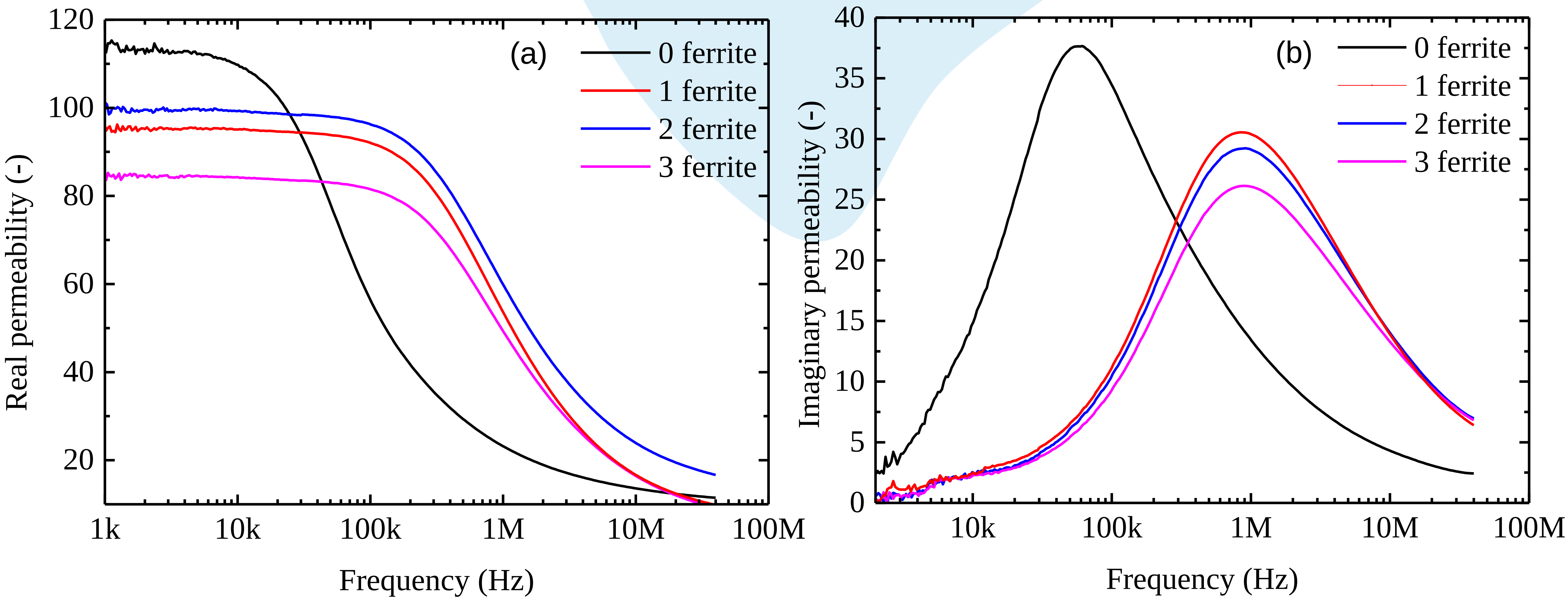





Shape of ASC100.29 iron particle
[Höganäs]
Coating
Mn-Zn ferrite
Iron particle
Compacting
Hydraulic press
Final
D=24 mm
d=18 mm
h=2.5 mm
Compacted SMC sample


(a)
(b)
SEM MAG: 100 x
500 µm
VEGA3 TESCAN
SAS KE
(c)
Mn-Zn FERRITE
IRON
UMV SAV
SEI
10.0kV
X1,000
10µm
WD 9.5mm

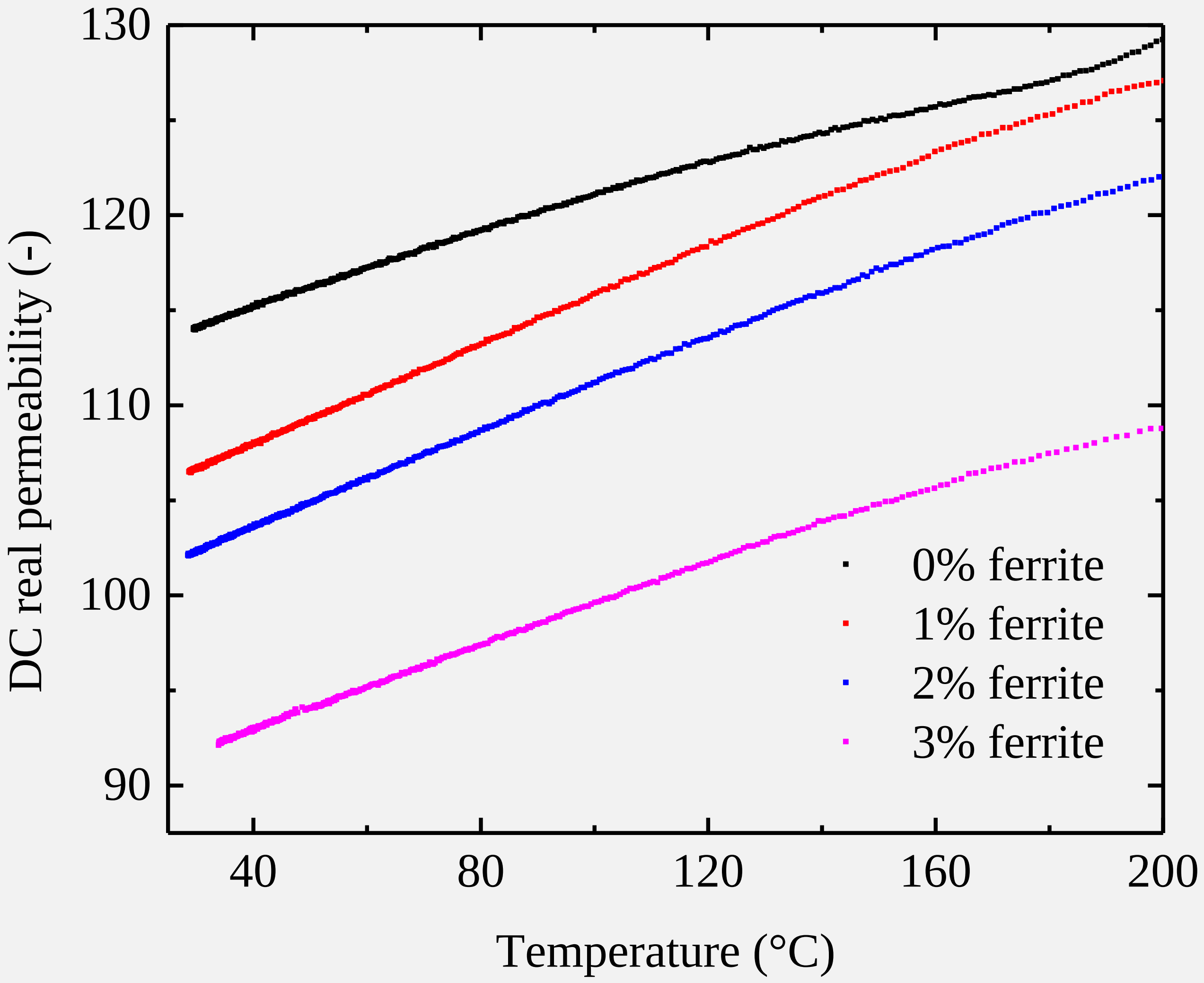

O
Fe
Mn
Zn
6 µm



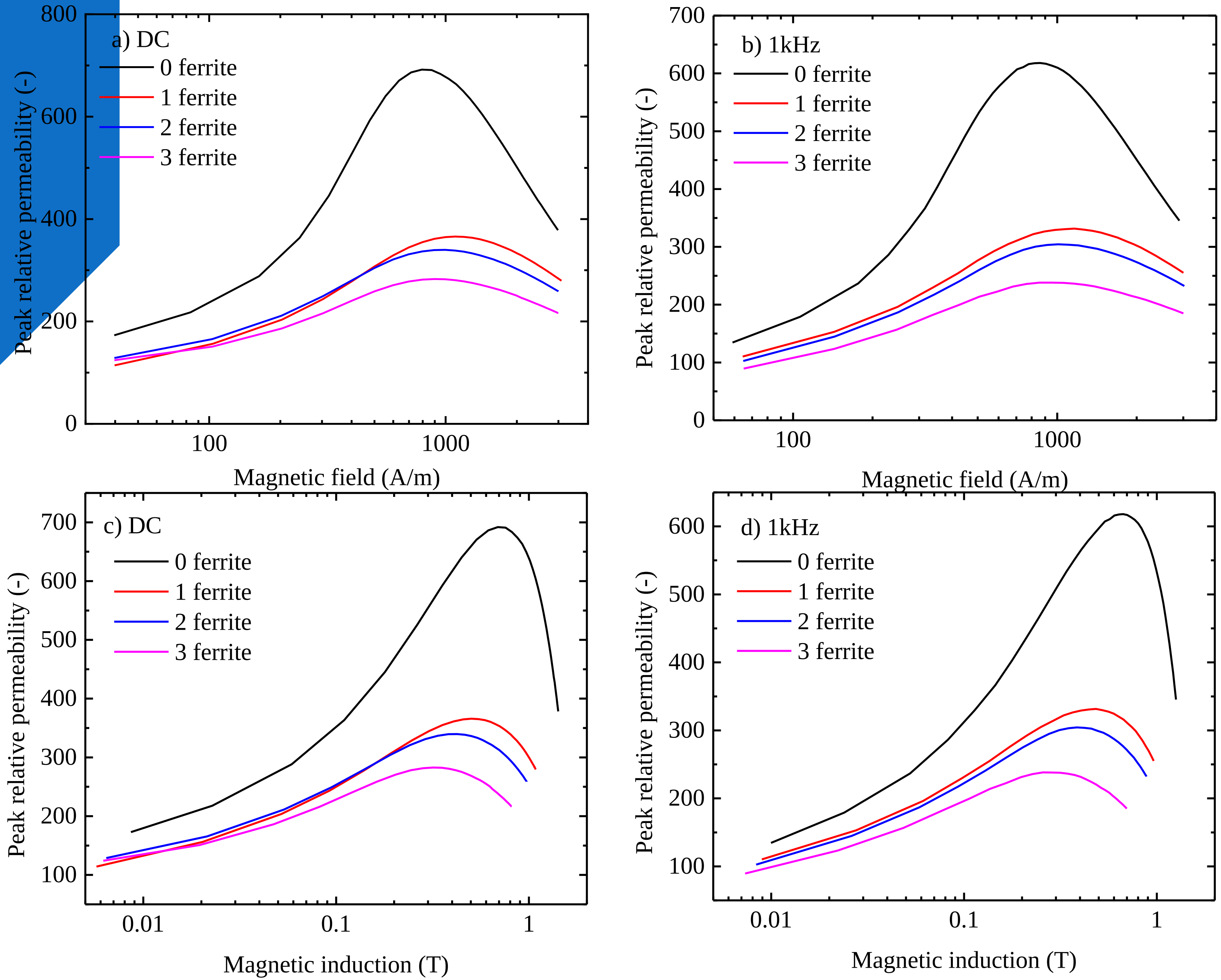
Frequency dependence of real (a) and imaginary (b) permeability of Fe/Mn-Zn ferrite composites with different content of soft magnetic ferrite coating.

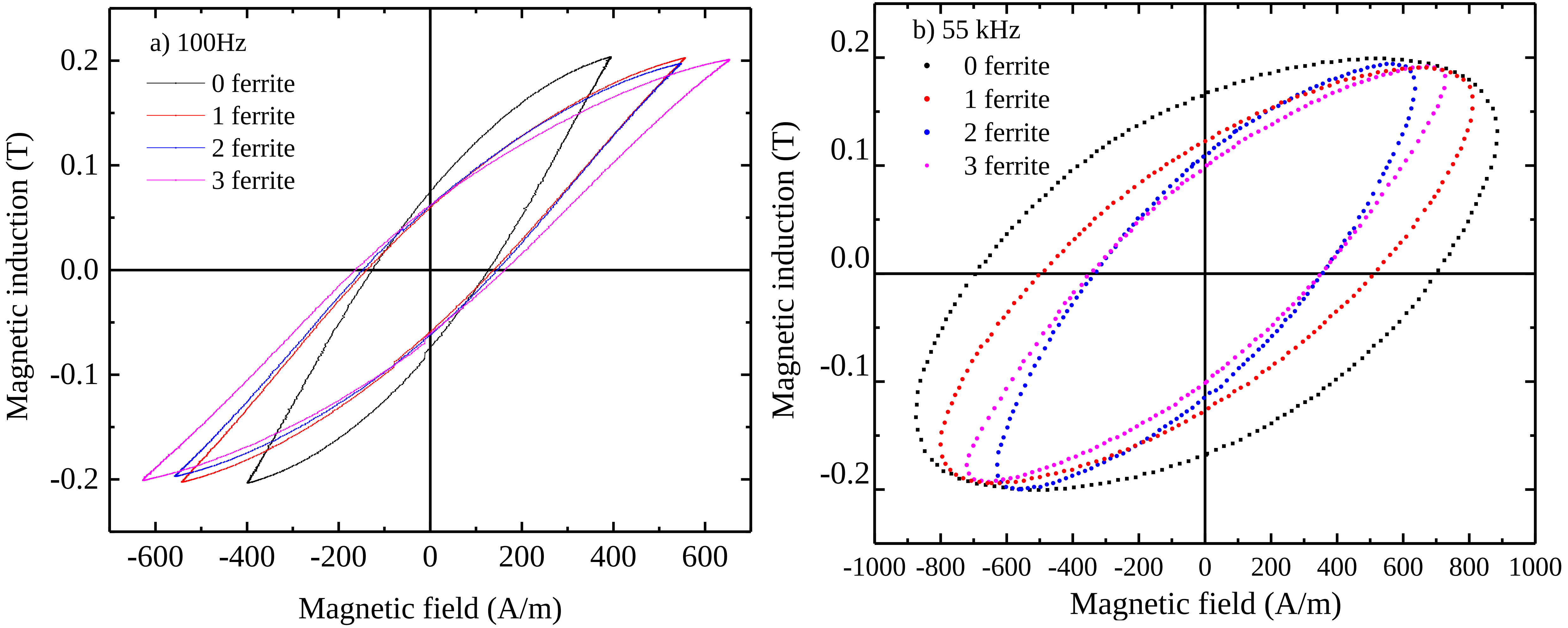
Sviatoslav Vovk, Ján Füzer, Samuel Dobák, Peter Kollár, Radovan Bureš, Mária Fáberová, Soft magnetic composite based on iron in sintered Mn–Zn ferrite matrix without non-magnetic coating, Ceramics International, Volume 49, Issue 18, 2023, Pages 30137-30146.

Temperature dependence of DC real component of permeability in Fe/Mn-Zn ferrite composites with different content of ferrite.



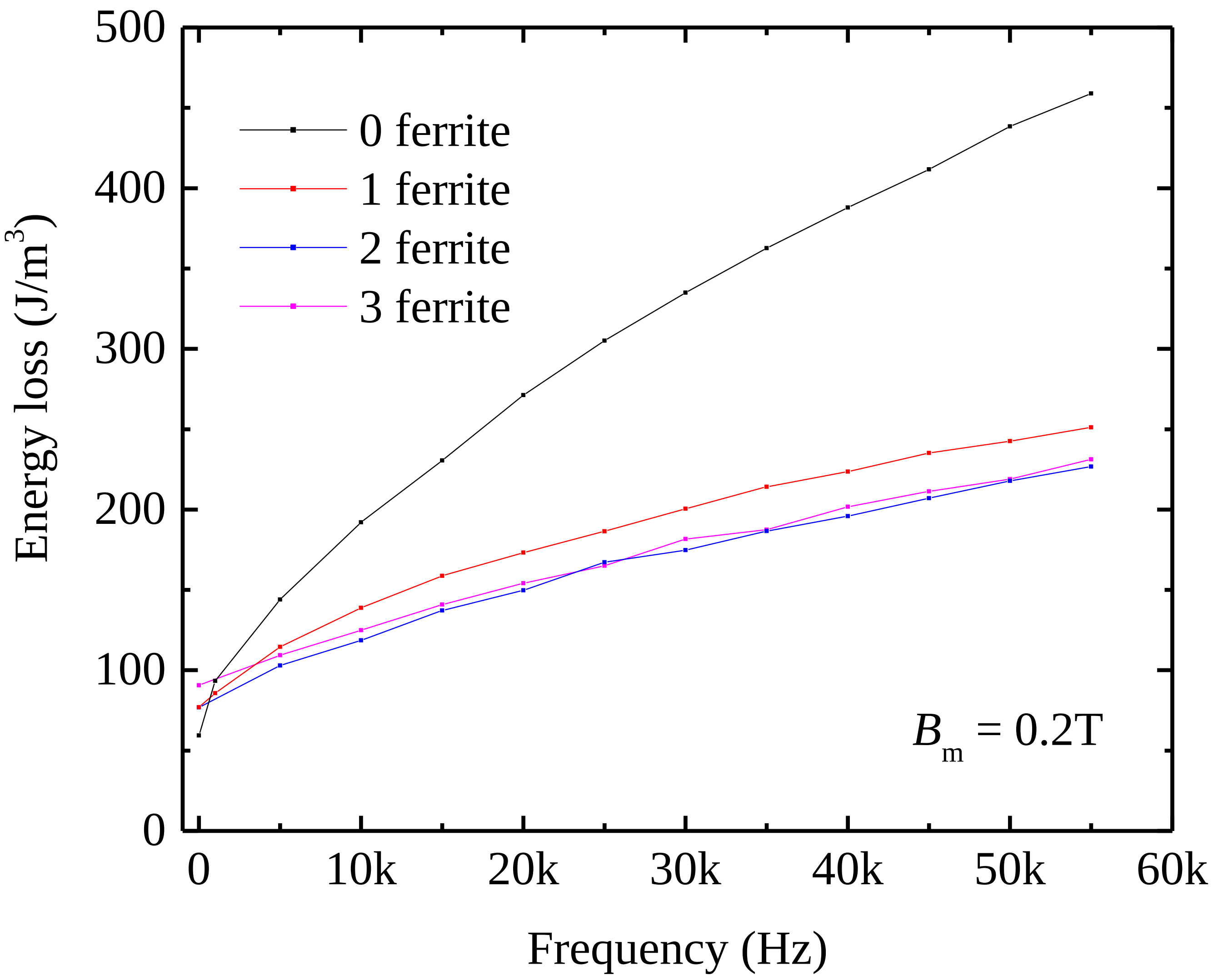
Peak relative permeability as a function of intensity of magnetic field H_m in the Fe/Mn-Zn ferrite composites (a) determined from DC hysteresis loop and (b) determined from hysteresis loop measured at 1 kHz





Hysteresis loops of the Fe/Mn-Zn ferrite SMC samples measured at (a) frequency 100 Hz at maximum induction 0.2 T, (b) frequency 1 kHz at maximum induction 0.2 T.

Energy loss as a function of frequency in the range 100 Hz - 55 kHz at maximum induction of 0.2 T of investigated Fe/Mn-Zn ferrite SMC samples.



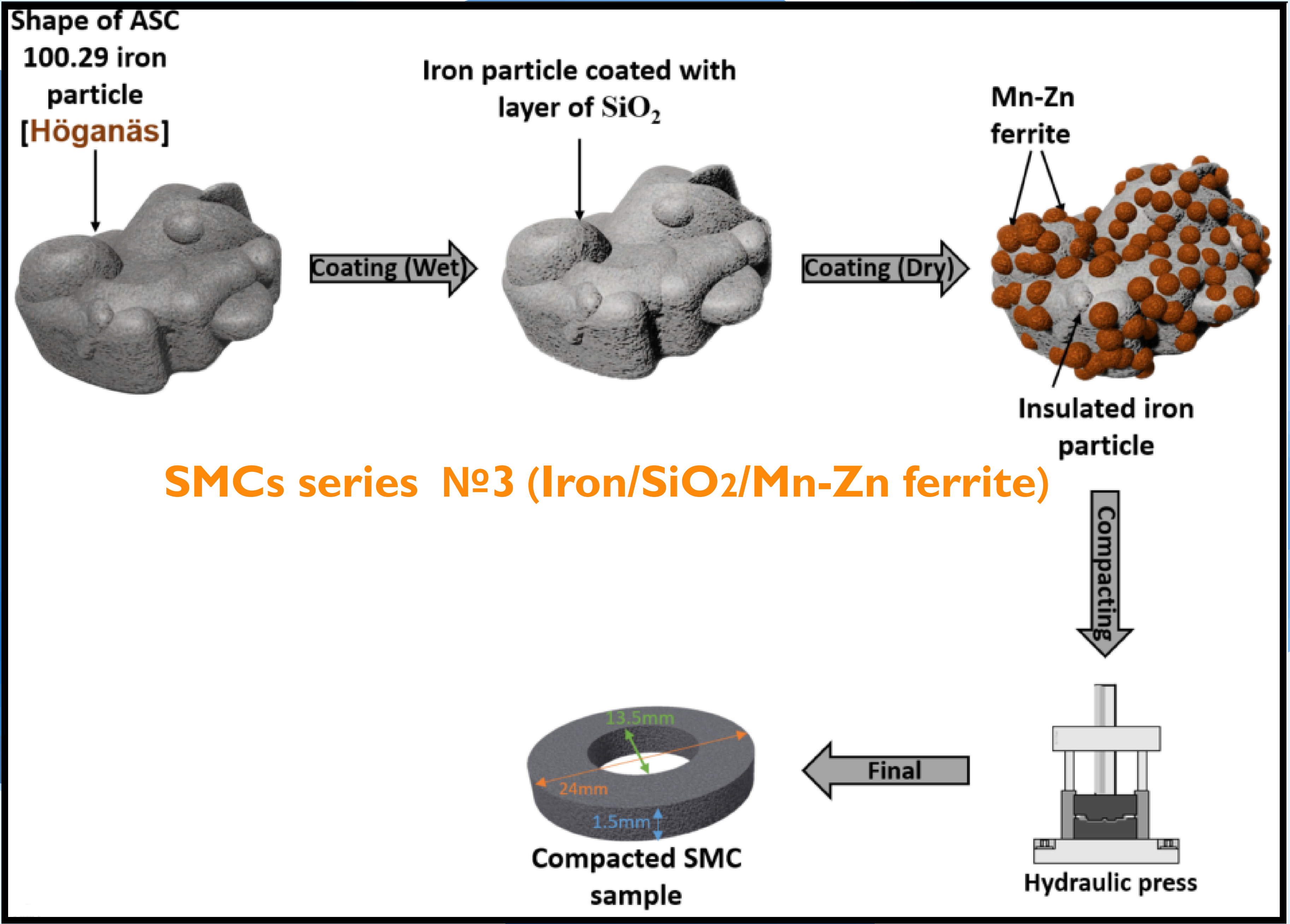
Conclusions: SMCs series №2

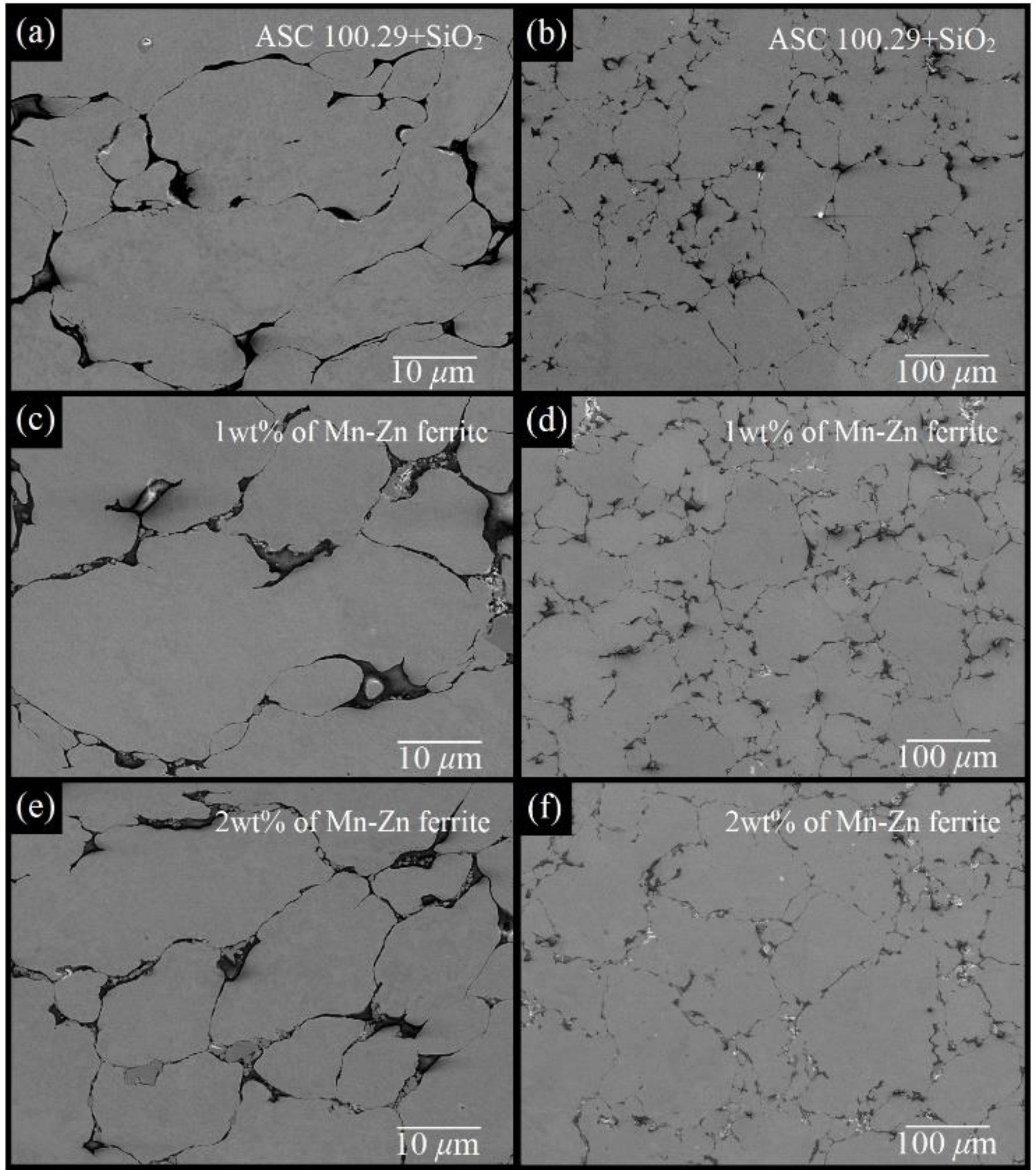
An advanced Mn-Zn ferrite-coated iron composite material was successfully prepared without additional non-magnetic binders

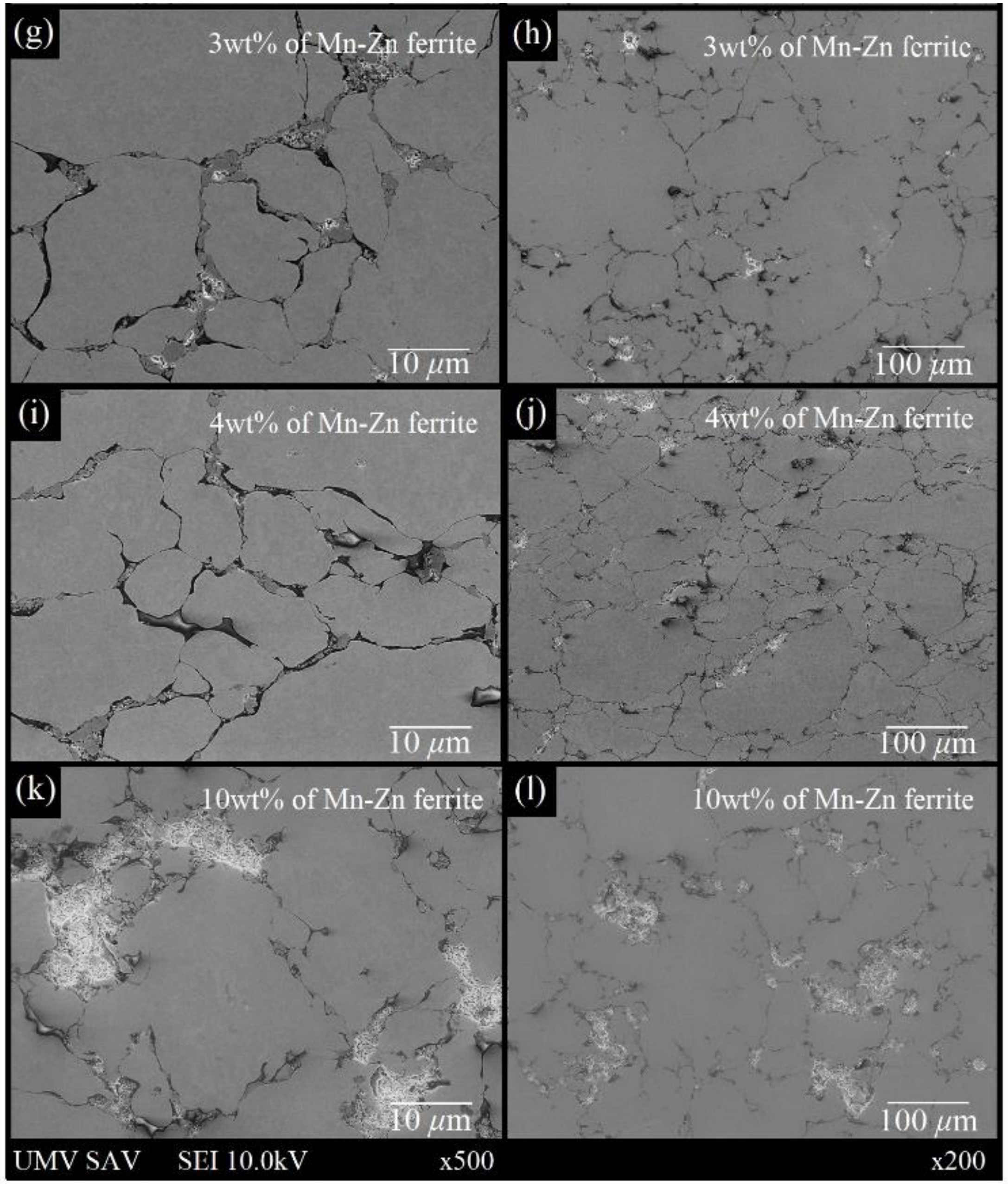
A non-conductive ceramic matrix, applied via dry mechanofusion, enhanced permeability values between 100 kHz and tens of MHz, shifted relaxation frequency higher, and reduced AC energy losses.

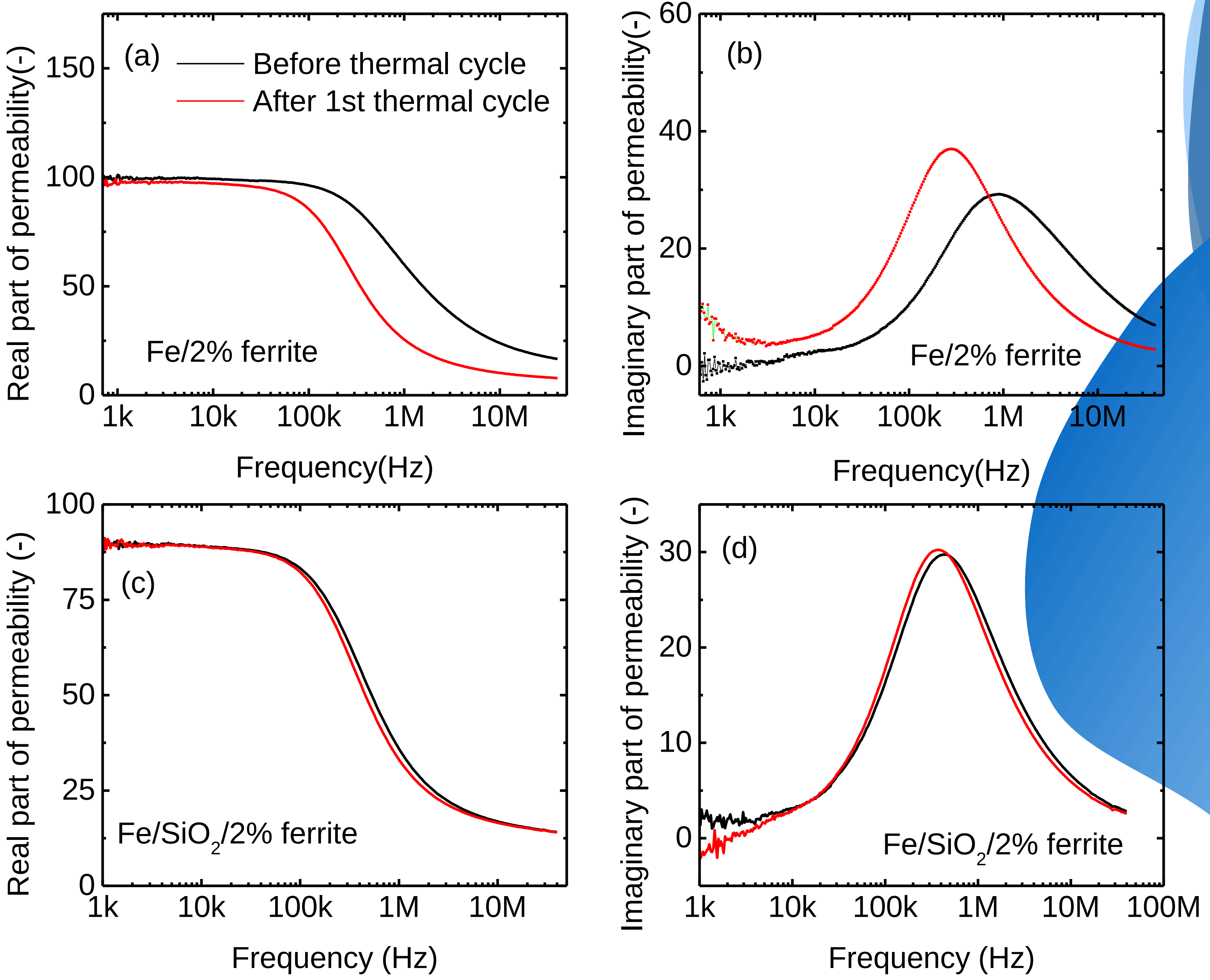
Stable magnetic permeability across frequencies and increased permeability with higher temperatures were achieved due to high Curie temperature ferrite coating.

The optimal ferrite amount, 1 wt%, yielded the highest permeability values at high peak inductions.

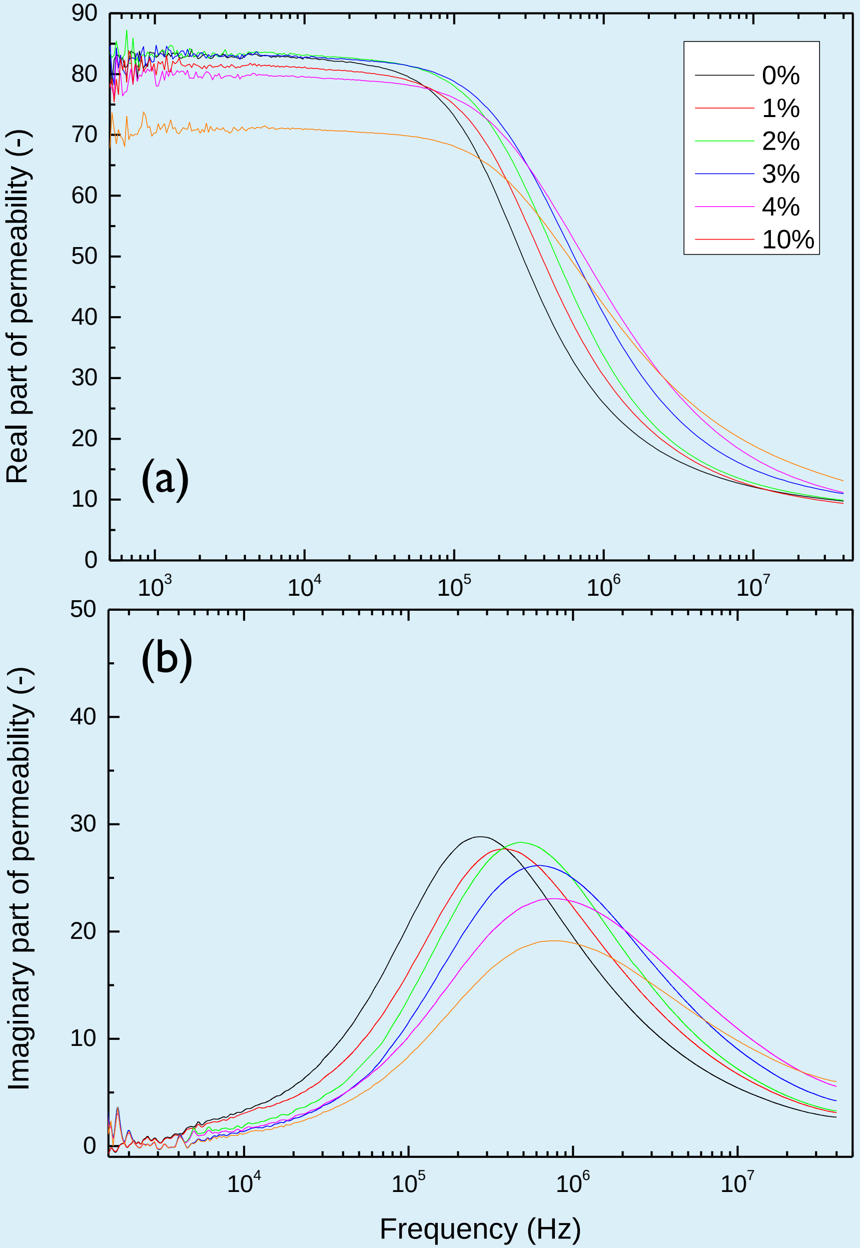

Shape of ASC
100.29 iron
particle
[Höganäs]
Iron particle coated with
layer of SiO2
Mn-Zn
ferrite
Coating (Wet)
Coating (Dry)
Insulated iron
particle
SMCs series №3 (Iron/SiO2/Mn-Zn ferrite)
Compacting
13.5mm
24mm
1.5mm
Final
Compacted SMC
sample
Hydraulic press

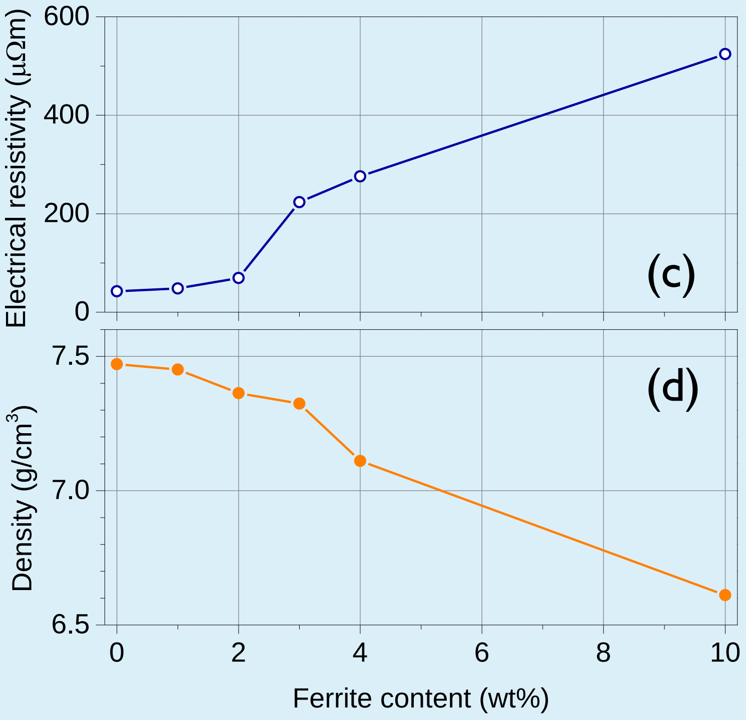

(a)
ASC 100.29+SiO2
10 μm
(b)
ASC 100.29+SiO2
100 μm
(c)
1wt% of Mn-Zn ferrite
10 μm
(d)
1wt% of Mn-Zn ferrite
100 μm
(e)
2wt% of Mn-Zn ferrite
10 μm
(f)
2wt% of Mn-Zn ferrite
100 μm


(g)
3wt% of Mn-Zn ferrite
10 μm
(h)
3wt% of Mn-Zn ferrite
100 μm
(i)
4wt% of Mn-Zn ferrite
10 μm
(j)
4wt% of Mn-Zn ferrite
100 μm
(k)
10wt% of Mn-Zn ferrite
10 μm
(l)
10wt% of Mn-Zn ferrite
100 μm
UMV SAV
SEI 10.0kV
x500
x200

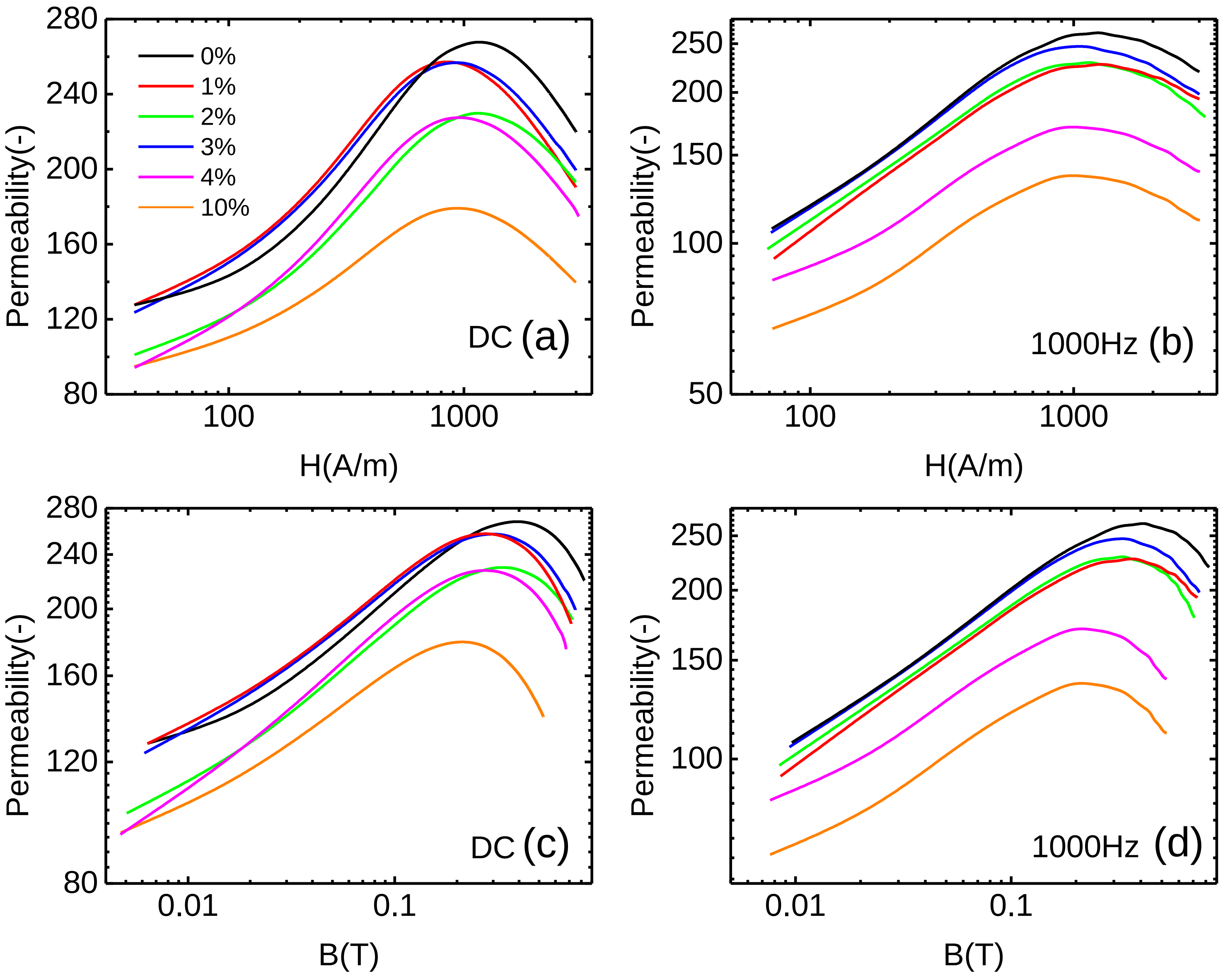


Effect of a thermal cycling on real and imaginary parts of complex permeability on Fe/Mn-Zn ferrite (a), (b) and Fe/SiO_2/Mn-Zn (c), (d) ferrite composites.



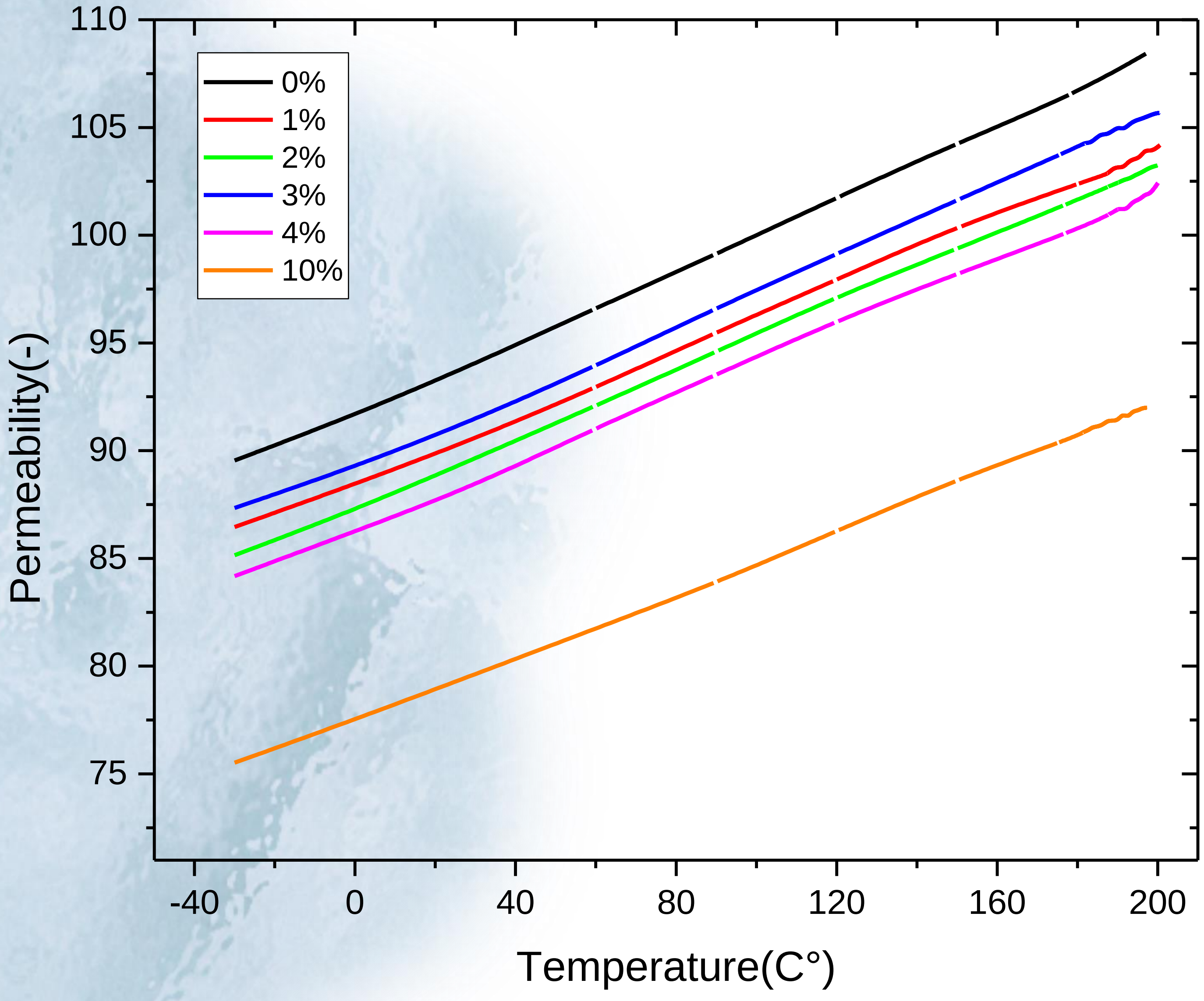


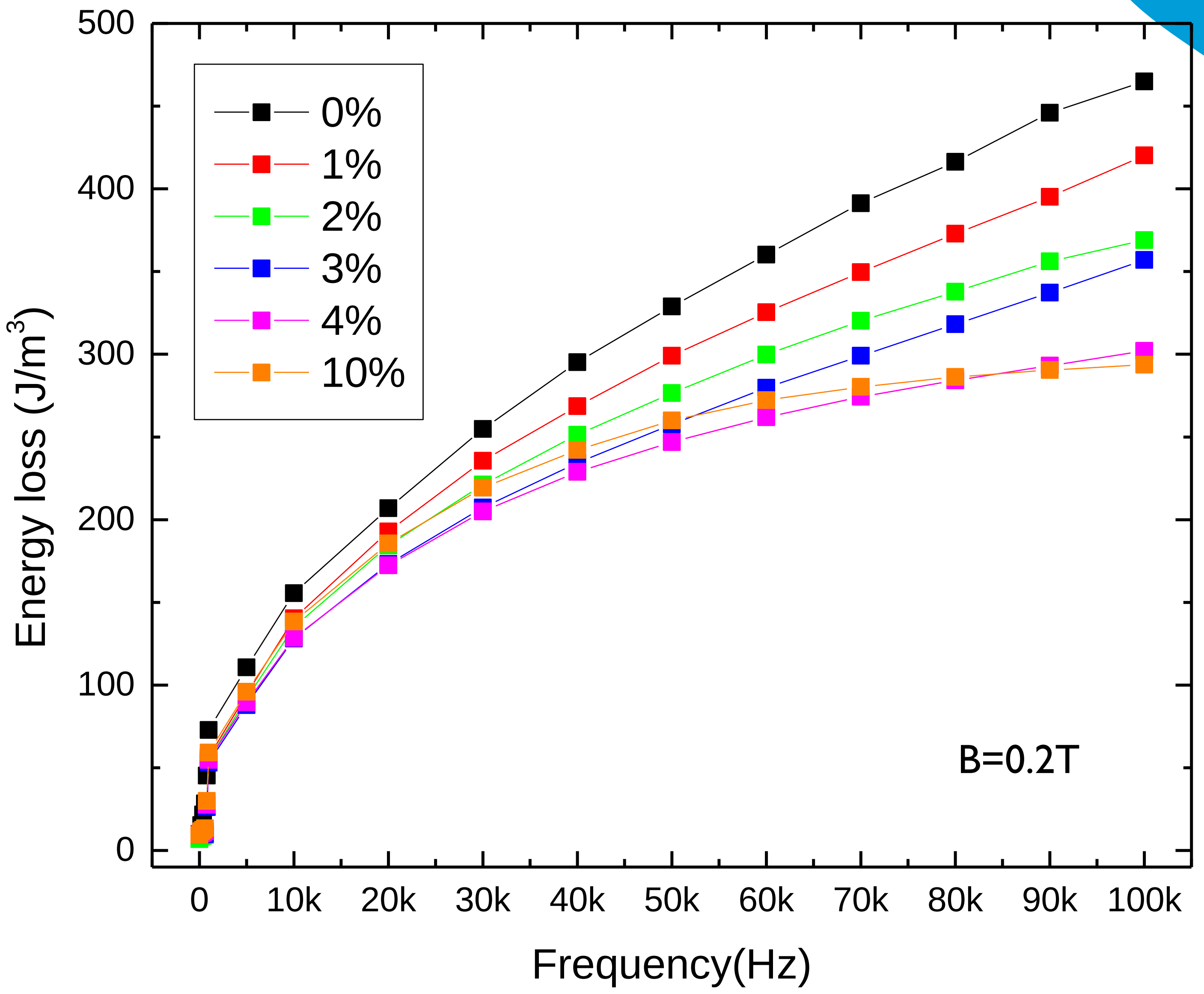
Frequency dependence of real (a) and imaginary (b) parts of complex permeability and electrical resistivity (c) and density (d) of Fe/SiO_2/Mn-Zn ferrite composites with different content of soft magnetic ferrite coating.



Peak relative permeability as a function of intensity of magnetic field H_m and of magnetic induction B_m in the Fe/SiO_2/Mn-Zn ferrite composites determined from DC and 1 kHz hysteresis loops.

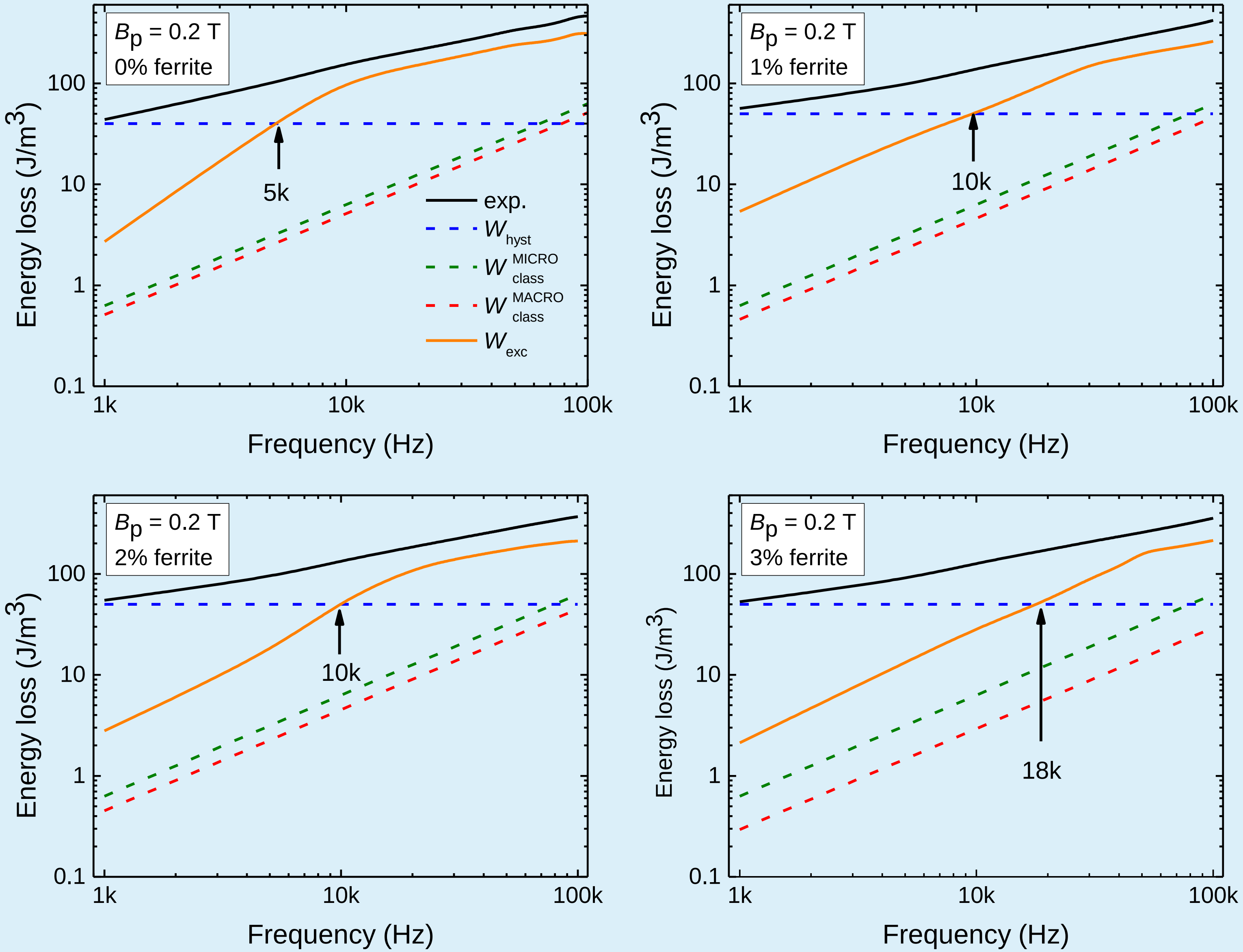
Temperature dependence of DC real component of permeability in Fe/SiO_2/Mn-Zn ferrite composites with different content of ferrite



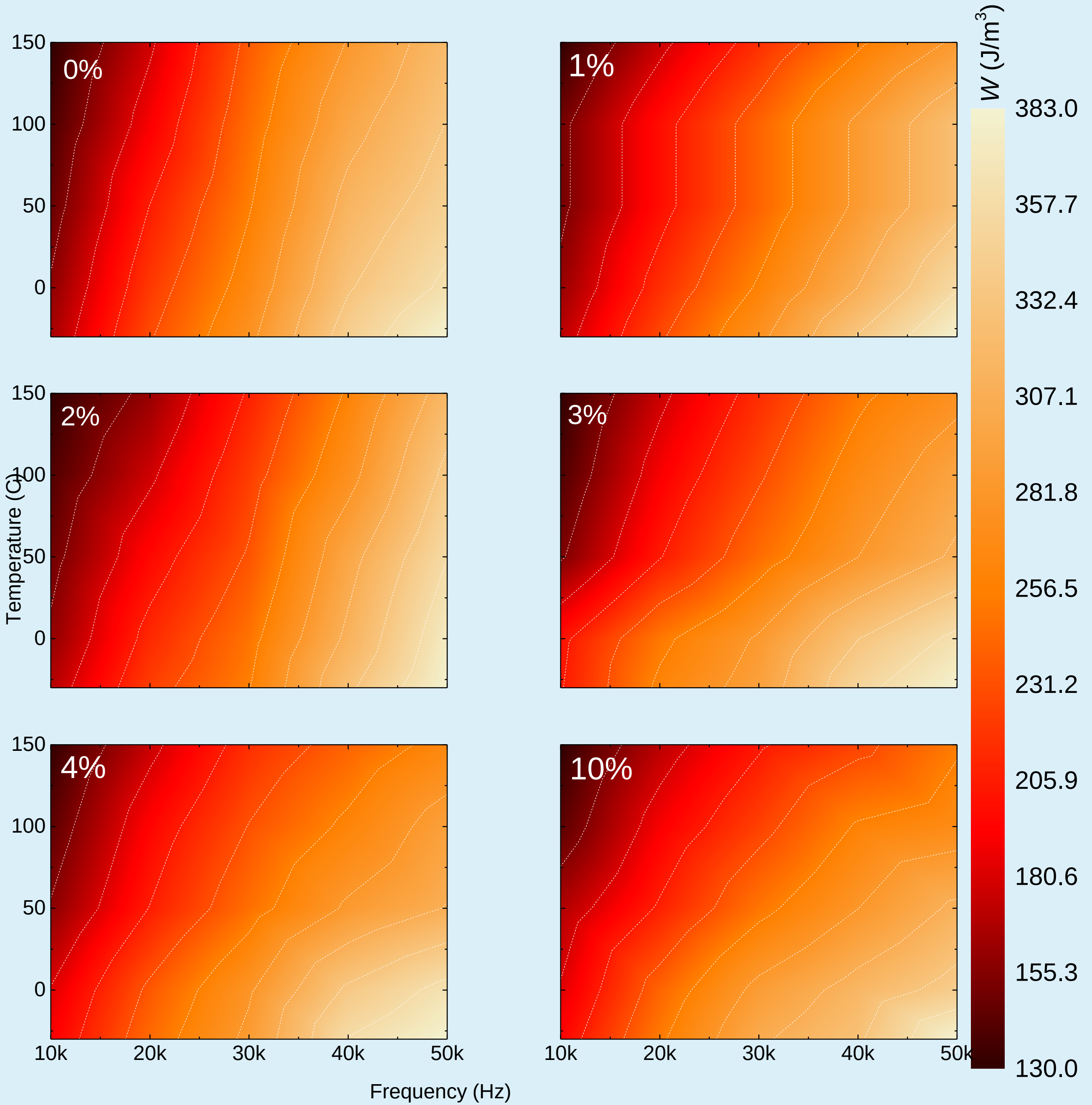


Energy loss as a function of frequency in the range DC - 100 kHz at maximum induction of 0.2 T of investigated Fe/SiO_2/Mn-Zn ferrite SMC samples.

Energy loss separation for Fe/SiO_2/Mn-Zn ferrite SMCs with 0%, 1%, 2% and 3% of ferrite in composition.



Frequency–temperature maps of energy loss in composites with 0 vol%, 1 vol%, 2 vol%, 3 vol%, 4 vol% and 10 vol% of ferrite measured at peak induction of 0.2 T.



Conclusions: SMCs series №3

Incorporating SiO_2 into an Fe/Mn-Zn ferrite composite enhances its thermal stability.

The new Fe/SiO_2/Mn-Zn ferrite composite maintains its structure from -30°C to 200°C, leading to increased permeability and reduced magnetic loss.

Although SiO_2 inclusion causes a drop in permeability, the consistency is retained after thermal cycling.

Optimal ferrite content in the composite lies between 2% to 4%, based on operational frequency needs.

Excess loss plays a significant role in mid-frequency range, suggesting a need to further study domain structure in SMCs.

LIST OF PUBLICATIONS

1. **Loss separation and thermal studies of Fe/SiO2/ferrite soft magnetic composites.**

   Sviatoslav Vovk, Samuel Dobák, Ján Füzer, Peter Kollár, Radovan Bureš, Mária Fáberová.

   Journal of Alloys and Compounds Volume 9455, 2023, Article number 169254.

2. **Soft magnetic composite based on iron in sintered Mn–Zn ferrite matrix without non-magnetic coating**

   Sviatoslav Vovk, Ján Füzer, Samuel Dobák, Peter Kollár, Radovan Bureš, Mária Fáberová, Vasiliki Tsakaloudi, V. Zaspalis.

   Ceramics International Volume 49, Issue 18, 2023 Pages 30137-30146.

3. **SiO2 layer as an important component for temperature stability of SMC based on iron and Mn-Zn ferrite**

   Sviatoslav Vovk, Ján Füzer, Samuel Dobák, Peter Kollár, Radovan Bureš, Mária Fáberová, Vasiliki Tsakaloudi, V. Zaspalis.

   Journal of Alloys and Compounds, *before submitting.*

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Soft magnetic composite based on iron in sintered Mn–Zn ferrite matrix without non-magnetic coating

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Research article

Loss separation and thermal studies of Fe/SiO_2/ferrite soft magnetic composites

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LIST OF ACTIVITIES

25th Soft Magnetic Materials Conference (SMM25, 2022, Grenoble, France)
Dependence of magnetic properties of hybrid SMC material on temperature – poster

Jarná škola doktorandov (JŠD, 2022, Liptovský Ján, Slovakia)
Dependence of magnetic properties of hybrid SMC material on temperature - presentation

The Joint European Magnetic Symposia (JEMS, 2022, Warsaw, Poland)
Influence of the temperature on electro-magnetic properties of hybrid SMC material - poster

Funkčné Kompozitne Materiály (FKM, 2023, Košice, Slovakia)
Novel soft magnetic composites with insulating layers formed by ferrite at elevated temperatures - presentation

Erasmus+
CERTH, Thessaloniki, Greece.
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