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X-ray magnetic circular dichroism study of $NiFe_2O_4$/ZrC composites

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1. Introduction:

Effective electromagnetic interference (EMI) shielding requires materials capable of attenuating incident electromagnetic (EM) radiation through reflection, absorption, or multiple scattering mechanisms. Conventional shielding materials predominantly rely on reflection, which necessitates high electrical conductivity and dense free charge carriers. However, reflection-dominated shielding often leads to undesirable secondary electromagnetic pollution due to re-radiation, thereby reducing overall shielding efficiency. Consequently, contemporary EMI shielding research has shifted toward absorption-dominated materials that dissipate EM energy via dielectric polarization losses, magnetic losses, and interfacial relaxation processes. Such materials are particularly crucial for advanced electronic systems, including medical imaging equipment (e.g., MRI scanners), aerospace electronics, and high-frequency communication devices. Metal-based EMI shields exhibit excellent electrical conductivity and shielding effectiveness; nevertheless, their practical implementation is hindered by several drawbacks, including susceptibility to corrosion, high density, elevated cost, and incompatibility with flexible, lightweight, or transparent electronic architectures. Polymer-based composites offer advantages such as low weight, corrosion resistance, and processability, yet they frequently suffer from inadequate mechanical strength, limited thermal stability, and moisture sensitivity. Similarly, transparent conducting oxides (TCOs) are constrained by relatively low electrical conductivity and restricted applicability in broadband EMI shielding [1]. Two-dimensional (2D) materials such as graphene and MXenes have emerged as promising alternatives due to their exceptional electrical conductivity, high mechanical robustness, large specific surface area, and tunable physicochemical properties. Since their discovery in 2011, MXenes derived from MAX phases have attracted significant attention for EMI shielding owing to their graphene-like layered morphology and mixed metallic, covalent, and ionic bonding. These unique bonding characteristics enable strong interaction with incident EM waves, promoting efficient absorption through multiple internal reflections, conduction losses, and interfacial polarization. Among them, Zirconium carbide (ZrC)-based MXenes are particularly attractive for EMI shielding, spintronic devices, and energy

storage applications due to their superior electrical conductivity, thermal stability, and structural hardness, as well as their strong hybridization capability with functional matrices. ZrC, a transition metal carbide with an exceptionally high melting point, is characterized by high hardness, moderate electrical and thermal conductivity, and excellent chemical stability. Unlike layered MXenes, ZrC possesses a non-layered rock-salt crystal structure with strong covalent bonding between zirconium and carbon atoms. When incorporated into a magnetic ferrite matrix, ZrC serves as an efficient EMI shielding component owing to its low density, appreciable electrical conductivity (~10^4 S m^{-1}), and outstanding thermal and oxidation resistance. Furthermore, ZrC exhibits a strong conduction-electron response and a high plasma frequency, which enhances EM wave reflection and promotes multiple scattering within the composite. Compared to conventional metallic fillers, ZrC offers superior long-term stability under harsh environmental and high-temperature conditions, making it a robust and reliable candidate for next-generation EMI shielding materials [1-7].

Integrating ZrC with $NiFe_2O_4$ achieves a synergistic balance of magnetic and conductive properties for enhanced EMI shielding [4]. ZrC boosts dielectric loss and reflection via its conductivity and interfacial polarization, while $NiFe_2O_4$ drives magnetic loss through domain wall resonance and natural ferromagnetic resonance [2,6]. Maxwell-Wagner polarization at the ZrC-$NiFe_2O_4$ interface enhances dielectric relaxation and charge hopping, converting electromagnetic energy into heat to minimize reflection pollution. The heterogeneous microstructure promotes multiple scattering for a broader attenuation bandwidth, with ZrC also tuning $NiFe_2O_4$ surface roughness, magnetic anisotropy, and crystallite size. Unlike oxidizable MXene or carbon fillers (e.g., graphene, CNTs), ZrC offers superior thermal stability, covalent bond integrity against electromigration/Joule heating, and densification for thermal conduction ideal for high-temperature applications in electronics, aerospace, and defence. Atomic-scale Zr-O interactions induce defect states and charge transfer, amplifying dipolar/interfacial polarization for broadband shielding, while improving corrosion resistance, mechanical hardness, and long-term endurance over pure ferrites.

2. Experimental sections:

$NiFe_2O_4$/ZrC composites were prepared via a modified sol–gel route using $NiFe_2O_4$ nanopowder and varying ZrC contents. Initially, ZrC nanoparticles were dispersed in 20 mL of ethanol and magnetically stirred at 80 °C for 2 h to obtain a homogeneous suspension with partial solvent evaporation. Subsequently, $NiFe_2O_4$ nanopowder was added and the mixture was further stirred for 10 min to ensure uniform mixing. The resulting suspension was dried below 70 °C to remove residual solvent, yielding fine composite powders, which were then subjected to structural, morphological, vibrational, and magnetic characterization to evaluate the $NiFe_2O_4$/ZrC interfacial interactions and their influence on composite properties.

The magnetic properties of $NiFe_2O_4$/ZrC composites were investigated using element-specific X-ray Absorption Spectroscopy (XAS) and X-ray Magnetic Circular Dichroism (XMCD) at the Ni and Fe $L_{2,3}$ edges. The measurements were performed at the undulator beamline BL-16A of the Photon Factory (KEK), Japan, enabling detailed element-resolved

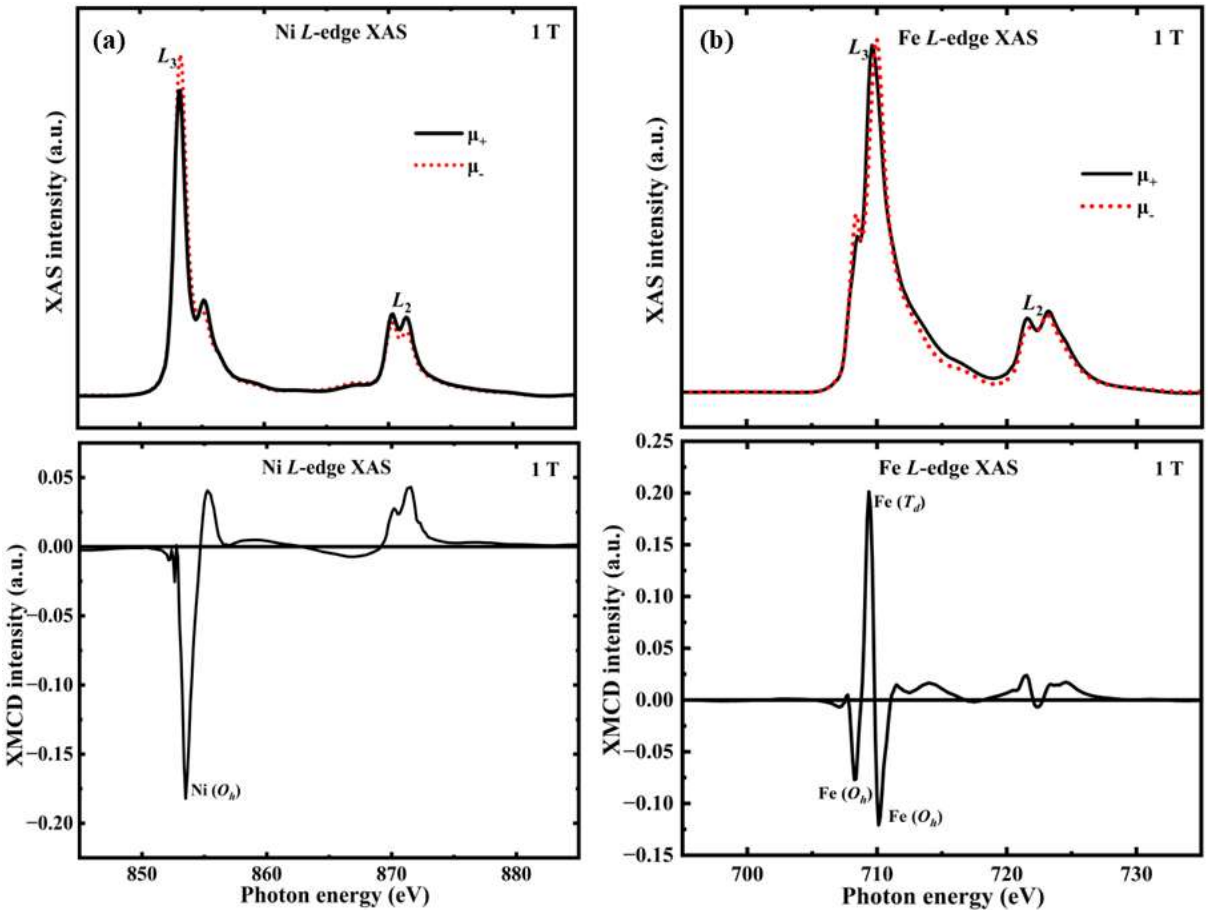

Figure 1: *Ni and Fe $L_{2,3}$-edge XAS and XMCD of $NiFe_2O_4$/ZrC composites.*

magnetic analysis. XMCD spectra were obtained from the difference in absorption coefficients (μ^+ and μ^-) for parallel and antiparallel photon helicities relative to the spin direction, using circularly polarised light. The magnetic field dependence of the XMCD intensity was analyzed to probe spin and orbital magnetic moments. The BL-16A monochromator provided an energy resolution of E/ΔE > 10,000 with a polarization degree of ~87 ± 4%. All measurements were conducted at 300 *K* under high-vacuum conditions (~10^{-9} Torr) in total electron yield (TEY) mode, with a probing depth of ~5 nm, using an out-of-plane geometry under a magnetic field.

3. Results and Discussions:

The Ni and Fe $L_{2,3}$-edge XAS, and XMCD spectra of $NiFe_2O_4$/ZrC composites are shown in the **Fig. 1**, providing element-specific electronic and magnetic information. The XAS spectra at the Ni and Fe *L*-edges show distinct L_3 and L_2 absorption peaks, which correspond to the $2p_{3/2} \rightarrow 3d$ and $2p_{1/2} \rightarrow 3d$ transitions. This confirms the trivalent Fe^{3+} and divalent Ni^{2+} oxidation states typical of inverse spinel $NiFe_2O_4$. When ZrC is included, the near overlap of the μ^+ and μ^- signals suggests a stable electronic structure with slight valence change.

The XMCD spectra show magnetic dichroism at the Ni and Fe edges, confirming their ferromagnetically ordered states. The XMCD signals for Ni^{2+} (O_h sites) and Fe^{3+} ions at T_d and O_h sites have opposing signs, indicating antiparallel spin alignment between *A*- and *B*-sublattices and inverse spinel ferrimagnetism. The strong Fe XMCD intensity suggests that the overall magnetic moment is dominated by Fe^{3+} ions, with a relatively smaller Ni contribution. The spectra show strong element-specific magnetic ordering in $NiFe_2O_4$/ZrC composites, with ZrC not disturbing the ferrimagnetic structure.

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