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POLYCRYSTALLINE Ni, Zn AND Ni-Zn FERRITE TARGETS FOR PLASMA LASER DEPOSITION TECHNIQUE

O. Caltun*, M. Feder[a], C. J. Liu[b]

"Alexandru Ioan Cuza" University, Faculty of Physics, 11 Blvd. Carol I, Iasi, 6600, Romania
[a]National Institute for Material Science, PO Box Mg-7, Bucharest, Romania
[b]Department of Physics, National Chang-Hua University of Education, Chang-Hua 50058, Taiwan, Republic of China

Ni, Zn and Ni-Zn ferrites targets to by used in pulsed laser deposition of thin ferrite films were prepared by a conventional ceramic technique. The properties of the ferrites were evaluated from XRD, SEM and EDX data. The electrical and magnetical characteristics of the investigated specimens are interpreted in terms of the role played by the sintering condition on the microstructure and on stoichiometry.

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Keywords: Spinel ferrites, PLD targets, Microstructure, Electrically and magnetically characteristics

1. Introduction

One of the most exciting challenges of the last thirty years of the past century in the solid state physics was the design and application of the new materials. The nanotechnologies, electronics, biophysics and computer science were the fields taking advantages from this competition. Thicker and thinner films carried out in different procedure were studied. One of the most used techniques of the last decay is the pulsed laser deposition (PLD) of thin films. The knowledge of the innermost processes was considered as limitations of the future development of this field [1]. The interest in PLD of thin films is justified by wide variety of the materials deposited by this technique [2]. From pure elements to multicomponent compound (up to six-element compounds) can be deposited. The stoichiometry of the target is reproduced in the films. The deposition "in situ" of the oxide materials without predeposition processes is an important advantage of the PLD technique, which is also simple, and low cost. Another advantage of is the deposition of the multi-layers thin films carried out by changing the gas pressure or the substrate temperature or the target.

One of the most important conditions in obtaining thin ferrite films with desired performances is the intrinsic quality of the target used in the plasma laser deposition process. In order to improve the quality of the PLD thin ferrite films this study was focused on the sintering process and complete microstructural, electrically and magnetically characterization of the Ni, Zn and NiZn ferrite targets.

2. Experimental results

A conventional ceramic process was used to prepare the targets. Three categories of target named Ni ferrite, Zn ferrite and Ni-Zn ferrite were produced starting from pure material, as follows:

* Was with Changhua University as Research Fellows in 2003, Corresponding author: caltun@uaic.ro

αFe_2O_3, containing 99.30 wt% Fe_2O_3 (as determined by titration with $KMnO_4$, after reduction in HCl solution of Fe^{3+} to Fe^{2+} with $SnCl_2$) and SiO_2 < 0.01% wt;

$NiCO_3.Ni(OH)_2.H_2O$, containing 60.02 wt% NiO (as determined by titration with EDTA);

ZnO, containing 99.20wt% ZnO (as determined by titration with EDTA) and PbO < 0.20 wt% (as determined by AAS).

The precursors were mixed in suitable proportions in a steel ball-mill for 3 hours using demineralized water in order to achieve respectively, the chemical formula $NiFe_2O_4$, $ZnFe_2O_4$ and $Ni_{0.5}Zn_{0.5}Fe_2O_4$. The dryed mixtures were presintered at 850 ^oC for 3 hours, in air. The X ray pattern of the presintered powder, presented in Fig. 1 show diffraction peaks specific for spinellic phase as well for "pure" precursors like αFe_2O_3 and ZnO. The targets structure was determined by X-ray diffraction using the SCINTAG DMS 2000 XRD equipment. The device uses the Fe K_α radiation (λ = 1.93604 Å) but in order to be rapidly related to the other data presented in referenced works the 2θ Fe K_α radiation were transformed by linear formula to the 2θ Cu radiation.

The presintered powder was milled in a steel ball-mill for 2 hours in demineralized water. The milled and presintered powders with BET specific surface of 4.3±0.3 m^2g^{-1} using polyvinyl alcohol (PVA) as a binder were dry pressed into disc shape having diameter and thickness of 28 mm and 6 mm, respectively. The green density of all specimens was 3.1±0.2 gcm^{-3}. After pressing, the discs were all sintered at 1100 ^oC two hours in air and cooled in nitrogen atmosphere with less 0.1 $\%O_2$. The samples densities were measured using Archimedes method.

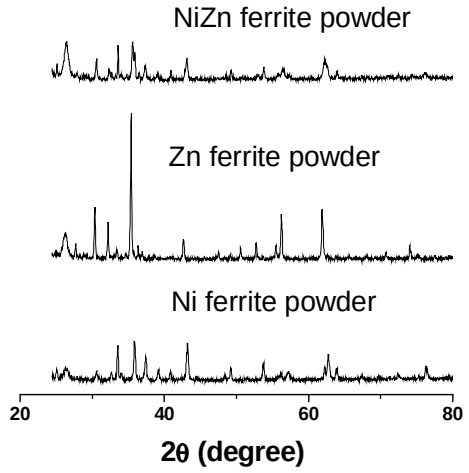


Fig. 1. The XRD patterns of the presintered powders.

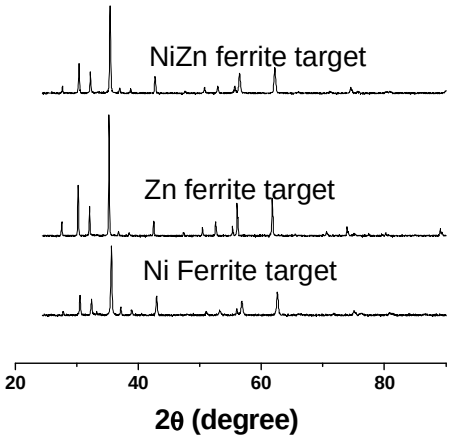


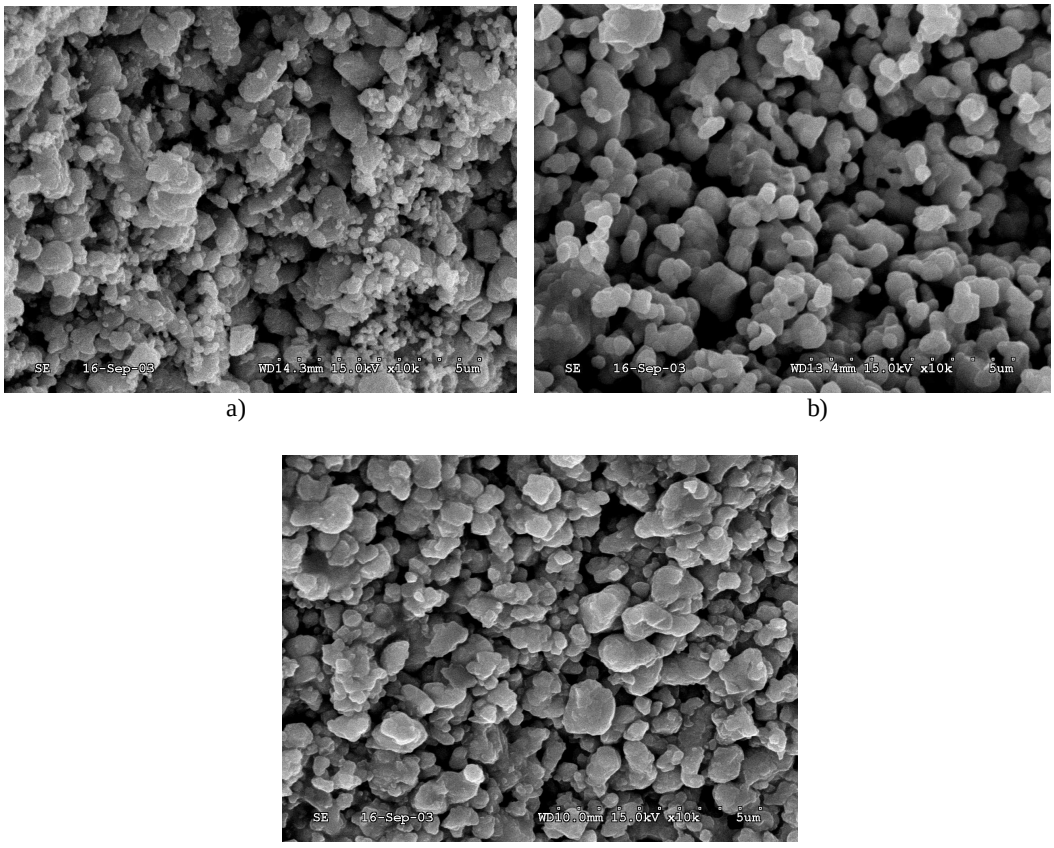
Fig. 2. The XRD patterns of the sintered targets.

After sintering process the XRD analysis of the targets as bulk material was performed in order to discriminate between the phase transformations. The XRD patterns presented in Fig. 2 straddle the high crystalline structure of the targets and the partial transformation of residual phases.

The microstructure and the composition of the sintered targets were evaluated from SEM and EDX measurements performed on a Hitachi S3000N scanning electron microscope, equipped with a Horiba electron microprobe. The microstructures in fracture are presented in Fig. 3. All the micrographs of the samples were centered to a non-uniform region with crystallites, vacancies and pores. The pictures stand for a uniform structure with grains dimension ranging in micrometer domain. The porosity of the sample is high due to the soft sintering condition. The EDAX data summarized in Table 1 confirm the stoichiometry of the samples.

Table 1. The composition of the ferrite targets measured by EDX analysis.

Element	Ni ferrite target		NiZn ferrite target		Zn ferrite target	
	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %
C K	1.59	5.14	1.56	4.52	1.75	5.24
O K	15.86	38.45	21.98	47.88	19.56	43.87
Fe K	55.13	38.29	50.68	31.63	52.65	34.52
Ni K	27.41	18.11	14.75	8.75		
Zn K			9.68	5.16	22.30	16.37



a) b)

c)

Fig. 3. The microstructure of the targets: a) Ni Ferrite; b) Zn ferrite; c) NiZn ferrite.

The DC resistance was measured by four probes method using a low noise multimeter Keithley 2010. The electrical conductivity of the samples was measured over the temperature range 25 – 650 ^{o}C. Plots of $log\rho/\rho_0$ versus ($10^3/Temperature$) are shown in Fig. 4. From this graph were calculated the activation energies for the ferromagnetic and paramagnetic regions. The data are summarized in Table 2.

Table 2. The resistivity at the room temperature for all targets and the activation energies.
ρ_0 DC resistivity at 25 ^{o}C, E_ρ Activation energies in ferromagnetic region at 40 ^{o}C, ΔE_ρ Differences between the activation energies $\Delta E_\rho = \Delta E_{\rho\ 40\ ^{o}C} - \Delta E_{\rho\ 440\ ^{o}C}$.

Sample	Ni ferrite	Zn ferrite	NiZn ferrite
$\rho_0(\Omega m)$	83.2	88.5	73.4
E_ρ (meV)	47	29	50
ΔE_p (meV)	22	3	4

From Fig. 4 and Table 2 one concludes that the DC resistivity for all targets has lower values than the values reported in [3]. This is due, in our opinion, to the special experimental conditions of sintering process, especially lower temperature than the conditions reported elsewhere [4]. The activation energies determinated from the *log*ρ vs. temperature graphs are in the same range with the experimental data reported in reference [5].

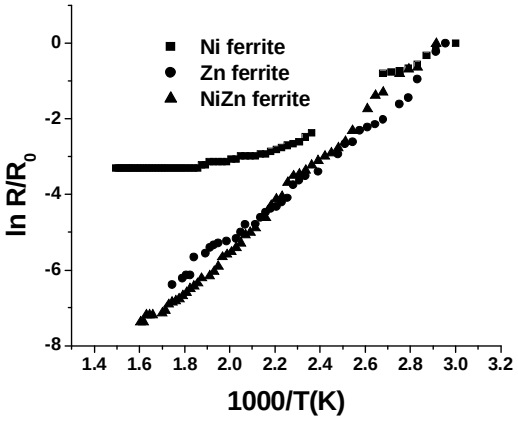


Fig. 4. The $\ln\rho/\rho_0$ versus (10^3/Temperature) (K^{-1}).

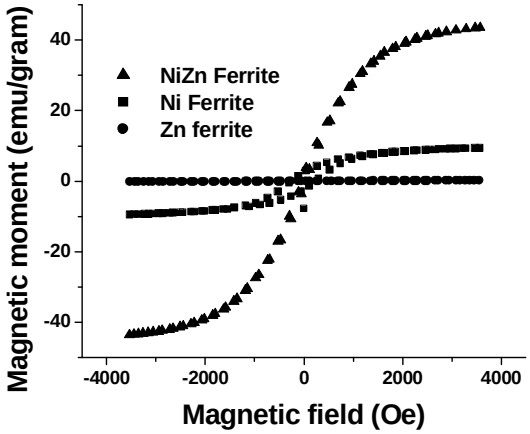


Fig. 5. Hysteresis loops for all the targets.

Magnetic measurements were performed using a vibrating sample magnetometer (VSM). The parameters of hysteresis loops presented in Fig. 5 demonstrate that the targets were sintered in soft conditions. Thus, from Fig. 5, Ni ferrite has a saturation magnetization of 10 emu/g, as compared to 50 emu/g, value presented as usual in [6].

3. Conclusions

Ni, Zn and Ni-Zn ferrites were prepared (as targets for PLD) by the ceramic method. High quality pellets were obtained, as resulted form the XRD, SEM, EDX and magnetic mesurements.

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