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Changes of Structural, Optical and Electrical Properties of Nickel-Manganite Ceramics Induced by Additional Mechanical Activation

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Introduction

NTC ceramics based on Mn, Ni, Co, Cu and Fe oxides have a wide application in electronic devices and sensors and are used for measuring temperature, temperature compensation, temperature control, voltage regulation etc.
These materials crystallize in the cubic spinel structure (general formula AB_2O_4) and nickel-manganite has an intermediate (partially inverse) cubic spinel structure.
In this study we have investigated the effects of additional mechanical activation of the starting powder mixture on the microstructure, far infrared (FIR) reflection spectra and electrical resistivity of sintered nickel-manganite samples.

Experimental

Nickel manganite powder was synthesized by the following procedure: calcination and vibratory milling from a mixture of MnO, NiO with 0.5 wt% CoO and Fe_2O_3.
The oxide mixture was additionally mechanically activated for different times – 15, 30 and 60 minutes in a planetary ball mill (a ball to powder mass ratio of 10:1).
Powders were subsequently pressed, then sintered at 1200°C for 1 hour.
Changes in the structural properties were analyzed using X-ray diffraction (XRD) Philips PW 1050 and scanning electron microscopy (SEM) JEOL JSM 6460 LV.
Room temperature far infrared optical reflectivity measurements were performed on a Brucker 113 V FTIR spectrometer.
The direct current resistivity (DC) was measured on a HP 4194A impedance/gain phase analyzer.

Results

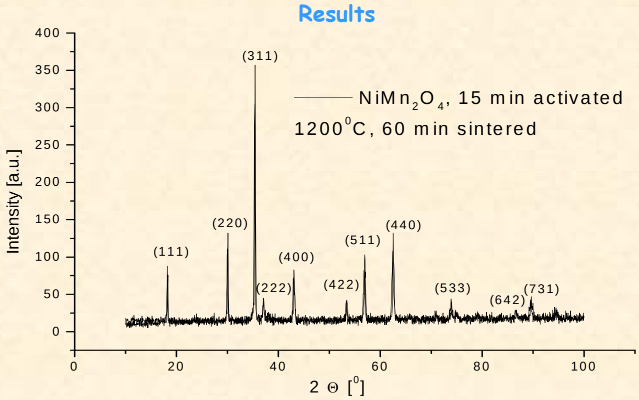


Fig. 1 XRD spectra obtained for $NiMn_2O_4$ mechanically activated 15 min. and sintered at 1200°C for 1 h

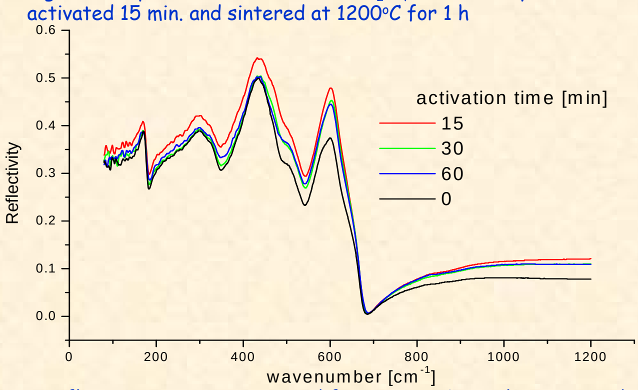


Fig. 2 Reflectivity spectra measured for NiMn2O4 samples activated for different times

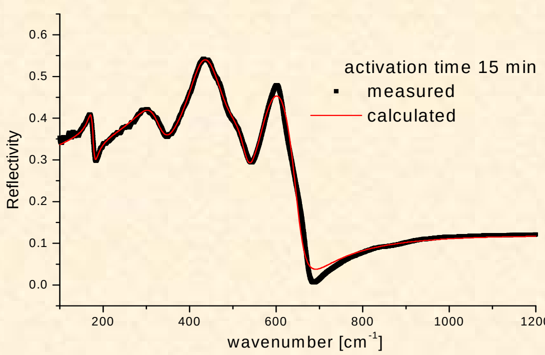
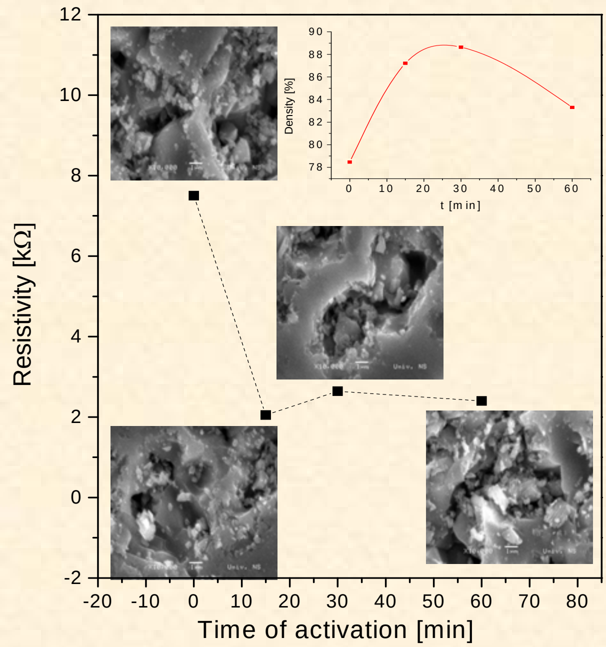


Fig. 3 Experimental (points) and calculated (line) IR reflectivity data for a $NiMn_2O_4$ sample mechanically activated for 15 minutes



Transversal and longitudinal frequencies (ω_{jTO}, ω_{jLO}) and damping factors (γ_{jLO}, γ_{jTO}) given in (cm^{-1}) calculated for $NiMn_2O_4$ for different activation time using the four-parameter model of coupled oscillators

Osc.	ω_{TO}	ω_{LO}	t [min]							
			0		15		30		60	
			γ_{TO}	γ_{LO}	γ_{TO}	γ_{LO}	γ_{TO}	γ_{LO}	γ_{TO}	γ_{LO}
1	174.5	178.1	11.9	10.3	12.9	11.8	11.7	11.0	13.7	12.9
2	239.6	294.4	319.6	459.8	562.9	1023.5	617.7	977.1	602.7	984.8
3	315.6	334.5	65.6	62.2	72.7	65.5	69.6	59.4	73.9	65.4
4	420.9	475.3	58.5	94.4	63.9	111.5	65.3	113.0	67.7	105.5
5	528.5	532.1	48.4	32.0	75.8	43.8	70.6	41.4	55.0	34.7
6	574.2	654.8	88.8	65.6	83.9	48.7	77.8	52.9	83.9	55.1

Conclusion

Short mechanical activation (15 min) of the powder prior to sintering lead to improved electrical conductivity (lower electrical resistivity) and increased sample density (lower porosity) that was reflected in a higher intensity of IR reflection peaks.

Longer mechanical activation resulted in higher agglomeration, slightly larger grains and higher porosity and also slightly increased dc bulk resisitivity that was also reflected in the intensity of IR reflection peaks!