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Ceramics International 32 (2006) 221–222

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Short communication

Sinterability of TiO_2-coated fine Ni powder

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Received 15 October 2004; received in revised form 8 December 2004; accepted 19 January 2005
Available online 26 April 2005

Abstract

In order to control the sinterability of fine Ni powder for internal electrodes of multilayer ceramic capacitors application, Ni particles were coated with TiO_2 by a homogeneous precipitation method. TiO_2 coating suppressed considerably the sinterability of Ni powder. TiO_2 powder additions also showed a suppressing effect on the sinterability of Ni powder, but in lesser extent if compared to the TiO_2 coating.

Keywords: Ni powder; Sinterability; TiO_2 coating; MLCC internal electrode

1. Introduction

Ni paste is widely used for the internal electrodes in multilayer ceramic capacitors (MLCC). The MLCC capacitance has been increased by increasing the number of thinner dielectric materials and by using thinner internal electrodes [1,2]. Thinner electrode films are realized by the use of small size Ni powder. In the simultaneous firing of multilayer ceramic capacitors, it is important that both dielectric materials and internal electrodes match their shrinkage behavior. This problem becomes more important when Ni electrode powder becomes finer.

In the present study, the effect of TiO_2 coating of Ni particles on the sintering behavior was investigated. The effect of mixing of TiO_2 particles with Ni particles was also examined for comparison purpose.

2. Experimental procedure

As-received 0.5 μm average size, 99.9% purity Ni powder (Wako Pure Chemical Ind.) was used. The TiO_2 powder (Ishihara Sangyo Kaisha) with particle size of 0.2–0.4 μm was produced by the chloride process.

TiO_2 coating of Ni particles was homogeneously precipitated using $TiOSO_4$ and urea as reactants [3]. $TiOSO_4$ and urea were dissolved in 200 ml dilute sulfuric acid below 10 °C. The concentrations of $TiOSO_4$ and urea were 0.01 and 1 mol dm^{-3}, respectively. After adding Ni powder, this solution was heated in a water bath at 60 °C for 1 h, at 70 °C for 1 h, and finally up to 80 °C. During reaction, the solution was stirred by a magnetic stirrer. The reaction product was filtered, washed, and dried. XRD of this product showed diffraction lines of Ni alone. The coating ratio was 0.05 g TiO_2/1 g Ni. Mixing of TiO_2 powder with Ni powder was done in ethanol by ultrasonic agitation.

Disks of 10 mm in diameter and ca. 2 mm thick prepared by uniaxial pressing at 2000 kg/cm^2 were sintered in quartz boat up to 600 °C, 800 °C or 950 °C at the heating rate of ca. 8 °C/min in a flow of nitrogen passed over titanium sponge. The densities were calculated from the size and weight of the disks.

3. Results and conclusions

SEM micrographs of the original Ni particles and of the TiO_2-coated Ni particles (Fig. 1) shows the uniform coating of TiO_2 on Ni particles. The thickness of the coating is estimated as 10 nm based on the ratio 0.05 g TiO_2/1 g Ni.

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0272-8842/$30.00
doi:10.1016/j.ceramint.2005.01.015

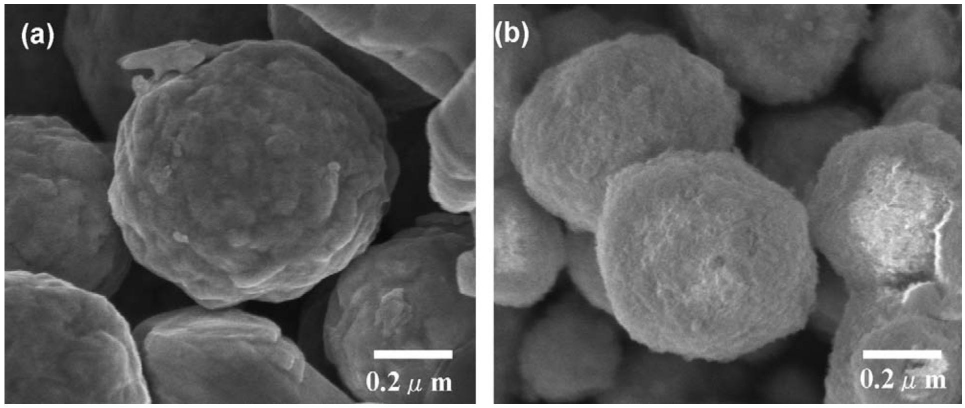

Fig. 1. SEM micrographs of (a) Ni and (b) TiO_2-coated Ni powder.

Table 1
Density and electrical resistance of sintered bodies

Ratio of TiO_2 to Ni (g/g)	Sintering temperature (°C)						
	Green density (%)	600		800		950	
		dr (%)	*R* (Ω)	dr (%)	*R* (Ω)	dr (%)	*R* (Ω)
Pure Ni							
0	51	64	0.003	93	0.003		
TiO_2 powder mixing							
0.025	51	66	0.002	85	0.007		
0.05	51	63	0.005	78	0.007		
0.075	51	60	0.004	74	0.004		
0.10	52	65	0.002	71	0.006		
0.15	52	56	0.006	68	0.007		
TiO_2 coating							
0.05	47	49	0.028	61	–	79	0.013
						85[a]	0.013

dr: relative density of sintered body heated up to sintering temperature at 8 °C/min and allowed for natural cooling; *R*: electrical resistance. These values should be taken as relative ones.
[a] Held for 1 h.

Table 1 summarizes the experimental results. The sintered density of Ni powder decreases by mixing of TiO_2 powder. The effect becomes remarkable at high sintering temperature. On the other hand, the suppression effect of TiO_2 coating is more remarkable than that of TiO_2 powder mixing. This may be due to the uniform coating of Ni particles by TiO_2.

The electrical resistance of sintered disks increases little by mixing TiO_2 powder in the additional level examined. On the other hand, the electrical resistance of sintered disk measured on TiO_2-coated Ni powder is somewhat higher, because of the uniform coating of Ni particle by TiO_2 which may reduce the number of Ni–Ni contacts in the sintered body.

Acknowledgements

The authors wish to thank M. Fujita, T. Yamamura, M. Noguchi, and N. Yamasaki for experimental assistance.

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