This article was downloaded by: [Tomsk State University of Control Systems and Radio]

On: 19 February 2013, At: 11:28

Publisher: Taylor & Francis

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered

office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



# Molecular Crystals and Liquid Crystals Incorporating Nonlinear Optics

Publication details, including instructions for authors and subscription information:

http://www.tandfonline.com/loi/gmcl17

# Preparation of Various Shape Superconducting Ceramics with Various Substrates by Electrophoretic Deposition Method

Nobuyuki Koura <sup>a</sup> , Hiromasa Shoji <sup>a</sup> & Arihiko Morita <sup>b</sup>

<sup>a</sup> Tokyo University of Science, Department of Industrial Chemistry, Chiba, 278, Japan

To cite this article: Nobuyuki Koura, Hiromasa Shoji & Arihiko Morita (1990): Preparation of Various Shape Superconducting Ceramics with Various Substrates by Electrophoretic Deposition Method, Molecular Crystals and Liquid Crystals Incorporating Nonlinear Optics, 184:1, 243-247

To link to this article: http://dx.doi.org/10.1080/00268949008031769

## PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <a href="http://www.tandfonline.com/page/terms-and-conditions">http://www.tandfonline.com/page/terms-and-conditions</a>

This article may be used for research, teaching, and private study purposes. Any substantial or systematic reproduction, redistribution, reselling, loan, sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae, and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand, or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

<sup>&</sup>lt;sup>b</sup> Nisshin Steel Co., Ltd., Hanshin R&D Laboratories, 5 Ishizunishimachi, Sakai, Osaka, 592, Japan Version of record first published: 22 Sep 2006.

Mol. Cryst. Liq. Cryst., 1990, vol. 184, pp. 243-247 Reprints available directly from the publisher Photocopying permitted by license only © 1990 Gordon and Breach Science Publishers S.A. Printed in the United States of America

PREPARAION OF VARIOUS SHAPE SUPERCONDUCTING CERAMICS WITH VARIOUS SUBSTRATES BY ELECTROPHORETIC DEPOSITION METHOD

NOBUYUKI KOURA, HIROMASA SHOJI, AND ARIHIKO MORITA\*
Tokyo University of Science, Department of Industrial Chemistry,
Chiba 278, Japan
\*Nisshin Steel Co., Ltd., Hanshin R&D Laboratories
5 Ishizunishimachi, Sakai, Osaka 592, Japan

#### Abstract

The YBCO superconducting ceramics with various substrates of various shapes were prepared by using the electrophoretic deposition method. The substrates were a Ag wire and an Al<sub>2</sub>O<sub>3</sub> ceramics plate. In the case of Al<sub>2</sub>O<sub>3</sub> ceramics, Ag was first coated on the ceramics by the electroless plating method. The superconducting properties were proved by the X-ray diffraction method, the diamagnetic measurement, and the resistivity measurement.

#### INTRODUCTION

In the preparation process of the superconducting oxides, the calcined oxides powder is usually pressed. After that, the oxide is heat-treated. If the electrophoretic deposition method is used instead of the press process, the superconducting ceramics in various shapes and sizes can be prepared<sup>1),2)</sup>. Moreover, various substrates can be used, if their surface is conductive. In the case of noncoductive one, it is beforehand coated with a metal.

#### **EXPERIMENTAL**

In the case of the silver substrate, the powder of  $Y_2O_3$ ,  $BaCO_3$ , and CuO were calcined, ground, then suspended in the acetone bath (100ml acetone,  $20mg~I_2$ ,  $0.3ml~H_2O$ , 1.0g~oxides) by an ultrasonic washing machine. On the electrophoretic deposition process, the electrolyte was the acetone bath, the W.E.(negative electrode) was Ag~substrate, and the C.E. was Pt~wire. The superconducting oxides coated material was heat-treated. In the case of nonconductive substrate, e.g.,  $Al_2O_3$  ceramics, at first it was mechanically etched and chem-

# <sup>244</sup> NOBUYUKI KOURA, HIROMASA SHOJI AND ARIHKO MORITA

ically etched<sup>3)</sup>, then coated with a metal, e.g., Ag by the electroless plating method<sup>4)</sup>. This Ag coated Al<sub>2</sub>O<sub>3</sub> ceramics was used as the W.E. for the above process. The conditions for the calcination and heat-treatment are; r.t. $\rightarrow$ (2h) $\rightarrow$ 880°C(10h) $\rightarrow$ (6h) $\rightarrow$ 300°C(0h) $\rightarrow$ (3h) $\rightarrow$ r.t., and r.t. $\rightarrow$ (3h) $\rightarrow$ 940°C(6h) $\rightarrow$ (6h) $\rightarrow$ 450°C(4h) $\rightarrow$ (3h) $\rightarrow$ r.t., respectively.

The superconducting properties were proved by the X-ray diffraction method, the diamagnetic measurement, and the resistivity measurement.

#### RESULTS AND DISCUSSION

#### Superconducting oxides

Amount of deposit of the superconducting oxides particle increased linearly with the cell voltage up to 1,200V as shown in Fig.1. At 1,200V, the deposit thickness was about 1,000µm.

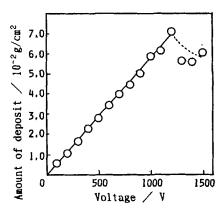


FIGURE 1 Relationship between the amount of deposition and the applied voltage.

The relationship between  $I_2$  content of the bath and the oxide deposit was obtained as shown in Fig.2. The peak value increased with increasing the cell voltage.

The oxides deposited sample was heat-treated. Two examples of an  $Al_2O_3$  ceramics and a silver coil substrates are shown in Photo 1. The coatings were dense and uniform. Their thickness was about  $200\mu m$ .

The coating was removed from the substrate, and pulverited. Then,

X-ray spectra was measured. The pattern coincided completely with that of the  $YBa_2Cu_3O_x(x=6.8\sim6.9)$ . The diamagnetic shift started at 93K.

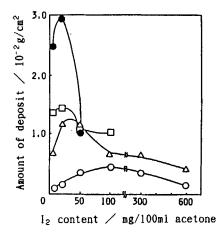


FIGURE 2 Relationship between  $I_2$  content in the bath and an amount of the deposit at various voltage. (Deposition time : 30 s)

Cell Voltage: ○ 50V. △ 150V. □ 300V. ● 600V

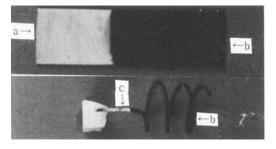


PHOTO 1 Photographs of  $Al_2O_3$  ceramics and Ag coil coated with the superconducting oxides.

(a):  $Al_2O_3$  ceramics.

(b): Superconducting oxides coating.

(c): Silver wire( ¢ 0.7mm).

Moreover, the resistivity of the coated superconducting material changed at about 90K from 2m $\Omega$  cm to zero(Fig.3). Zero means smaller value than  $0.1\mu\Omega$  cm.

(2) Electrophoretic deposition mechanism
When the superconducting oxides powder was added to the acetone

bath, the conductivity became about half (Fig.4). This decreasing may be attributed to an adsorption of  $\mathrm{H}^+$  onto the oxides particle surface. The bath conductivity increased significantly with increasing the  $\mathrm{H}_2\mathrm{O}$  content up to 3%, and increased gradually up to 10%.

On the other hand, a zeta-potential of the oxides particle in the

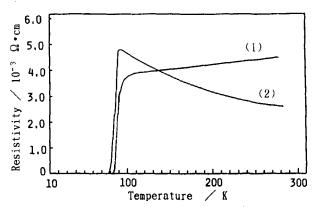


FIGURE 3 Resistivity of the sample from (1)the acetone bath and (2)the acetylacetone bath.

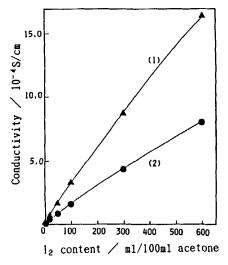


FIGURE 4 Relationship between  $I_2$  content in the bath and a conductivity of the bath (1)without, (2)with superconducting powder.

bath was about +60mV. This fact also indicates that the H<sup>+</sup> ion adsorbs onto the oxides particle. Therefore, the mechanism shown in Fig.5 consequently decided. Acetone generates the H+ ion according to the keto-enol reaction and the iodine addition reaction. This H<sup>+</sup> ion adsorbs onto the oxides particle. The charged particle deposits on a negative electrode by a high voltage electrolysis.

## (3) Improvement of the electrolyte

On the electrophoretic deposition process, an acetone bath has been used. But, acetone is flammable. Then, safe solvent was studied.

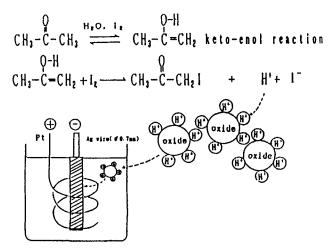


FIGURE 5 Electrophoretic deposition of oxide powder.

An acetylacetone bath was found. A uniform, thick, and dense oxides coating was obtained. However, the  $T_{\rm C,end}$  decreased remarkably to 25K. Then, the drying process of the electrophoretically deposited sample in vacuo, the pre-heating process at  $100^{\circ}{\rm C}$ , and the  $0_2$  flowing during the heating process were devised. As the results, the  $T_{\rm C,end}$  became about 80K as shown in Fig.3.

#### REFERENCES

- 1. N.Koura, et al., Denki Kagaku, 56(3), 208(1988)
- 2. N.Koura, et al., <u>J.Surf.Fin.Soc.Japan</u>, <u>40</u>(7), 819(1989)
- 3. N.Koura, et al., <u>J.Met.Surf.Fin.Soc.Japan</u>, <u>36</u>(5), 182(1985)
- 4. N.Koura, et al., ibid., 37(3), 131(1986)