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Determination of the Metallic Elements in high Tc Superconductors M-X-Cu-O (M=Ba,Sr, X=Y,La) System by Isotachophoresis Method and X-ray fluorescence method

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Abstract Molar ratios of oxide-superconductor powders which are prepared by a coprecipitation method and a solid reaction method have been determined by isotachophoresis method using a leading solution with potassium acetate and a terminal solution with tris(hydroxymethyl)aminomethane.

INTRODUCTION

Molar ratios of the oxide-superconductor powders[1,2] which are prepared by a coprecipitation method and a solid reaction method frequently deviate from Y:Ba:Cu = 1:2:3 molar ratio, however it needs the accurate molar ratio of Y, Ba, and Cu in oxide-superconductors for having high Tc. The precise composition analysis of the oxide-superconductors is very important for making high Tc superconductors.

The composition analysis which have ever been reported in journals and at the annual meeting of Japan Society, of Applied Physics, and of Analytical Chemistry, were Induced-couple-plasma-emission spectrometric(ICP-AES) method[3], X-ray fluorescence (XRF) method[4], Electron-prove-micro analysis (EPMA) method[5], substiometric radio-chemical analysis for metallic elements

(Y,Ba,and Cu), and radio isotopic method[6] and gas analysis for oxygen. Though ICP-AES method is good one, it needs rather much solutions. As XRF and EPMA methods are surface analysis, it does not give always real bulky compositions. Radiochemical method are difficult one in general. In the contrary with the above methods, isotachophoresis(IP) method[7] is simple one and does not need much solution. Further, because simultaneous determination of Y, Ba, and Cu is possible, the analysis can be done in a short time.

In this paper, we have investigated about a simultaneous determination of Y, Ba, and Cu in the superconductors by the IP method. The method has been successfully applied to determine simultaneously Y, Ba, and Cu in oxide superconducting materials.

EXPERIMENT

The yttrium, lanthanum, barium, strontium and copper in high Tc superconductors M-X-Cu-O(M=Ba,Sr, X=Y,La) system have been determined by the capillary tube IP method(IP-1B made by Shimazu Co.Ltd.). A leading solution and a terminal solution are 0.01 M potassium acetate solution including 0.05 % polyvinyl alcohol (pH=5.40) and 0.01 M tris(hydroxymethyl)aminomethane (pH=5.0). This method was applied to $YBa_2Cu_3O_{7-X}$ samples (Tc 90K) prepared by the co-precipitation method and the solid reaction method.

The isotachopherogram of the sample is shown in Fig.1. Small amounts (2 - 6 microliters) of the sample solution were used for the isotachophoretic measurements and another part of the solution was used for the ICP-AES method.

The IP measurement conditions are shown in Table 1.

Table I Experimental Conditions

1. Leading solution	0.01 M Potassium acetate
	0.05 % polyvinyl alcohol(pH=5.40)
2. Terminal solution	0.01 M Tris(hydroxymethyl)aminomethane
	(pH=5.00)
3. pH control	potassium acetate and acetic acid
4. Migration current	100 μA (20 min)
5. Capillary length	20 cm
6. Inside diameter	0.5 mm
7. Oven temperature	20 °C
8. Sampling weight	0.01 - 0.1 g
9. Dissolution of samples	CH_3COOH, HNO_3 or HCl solution

RESULTS AND DISCUSSION

The composition ratios of the samples made by coprecipitation method and the solid reaction method were Y:Ba:Cu=0.95:1.97:3.00 and Y:Ba:Cu=0.99:1.98:3.00, respectively. The analytical results in this method were agreed with the results of the ICP-AES method and the XRF method. The repeatability of the determinations by the IP method was ± 0.01.

Calibration curves of Y-Ba-Cu system, Y-Sr-Cu system, and La-Ba-Cu system were linear and the zone length of each element was proportional to the concentration of each element.

The composition ratios of high Tc oxide superconductors prepared by coprecipitation method and solid reaction method are shown in Table II. The accuracy of results were agreed within 0.02 - 0.08 of the starting composition, and were agreed with the results of X-ray analysis and ICP-AES method.

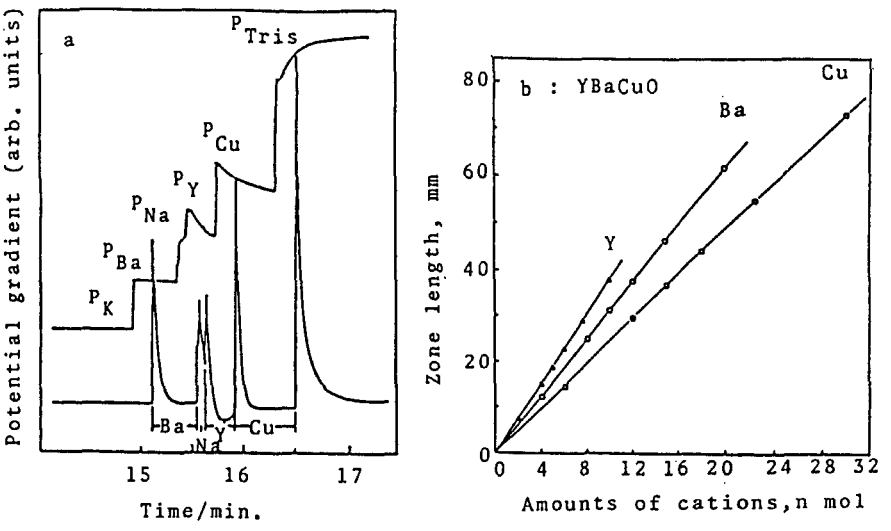

Fig.1 Isotachopherogram(a) and Calibration curves(b) of Y, Ba, and Cu in the CH_3COOK-Tris. system.

Table 2 The results of actual samples

No.	preparation method	condition	analytical method	results Y :	Ba :	Cu	Tc K
1.	coprecipitation	oxalate	starting#	1.0	2.0	3.0	
			IP	0.97	2.02	3.00*	
2.	coprecipitation	600°C 900°C	starting	1.0	2.0	3.0	
			IP	0.95	1.97	3.00*	88.0-77.5
3.	oxide mixture	900°C 930°C	starting	1.0	2.0	3.0	
			IP	0.99	1.98	3.00*	92.0-91.3
			ICP-AES	1.02	2.07	3.00**	
4.	coprecipitation	930°C	starting	1.0	2.0	3.0	
			IP	0.92	1.92	3.00***	80.1-74.0
			X-ray	0.93	1.97	3.00	
5.	oxide mixture		starting	1.72	2.57	3.0	
			IP	1.69	2.57	3.00***	

* 2 times average, ** 10 times average, *** 5 times average,
# starting composition

This method has the following promises; 1. amounts of sample solutions are only 2 - 6 micro litter, 2. Y, Ba, and Cu can determined simultaneously, 3. procedure for the determination is simple, 4. repeatability was within 2%, 5. whole determination was able to perform within 1 hour, 6. all samples in the state of powder, crystal, and thin layer containing Y, Ba, and Cu, are able to determine, simultaneously.

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