APPLICATION NOTE

Structural Evaluation of Niobium Hydroxide and Niobium Carbide (Reference Catalysts) by Gas Adsorption

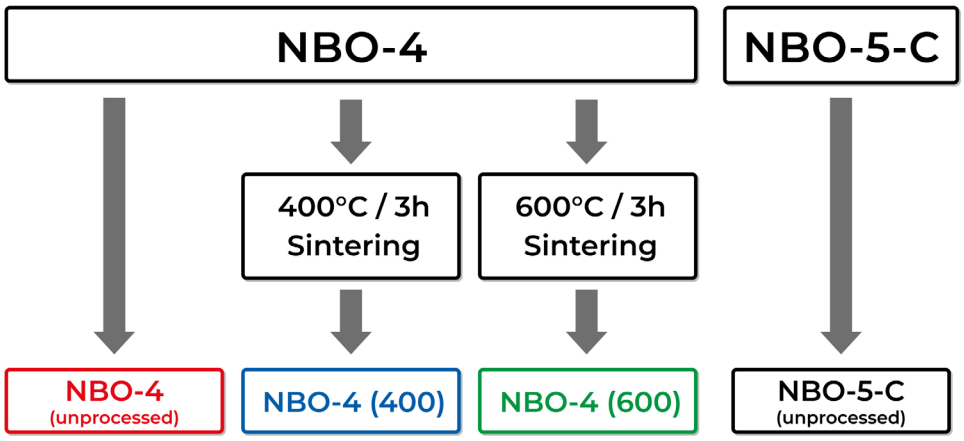
(Specific Surface Area, Pore Distribution) and He Gas Substitution (True Density Evaluation)

Overview

True density measurement using nitrogen adsorption/desorption isotherm measurement and He gas substitution was carried out on niobium hydroxide (reference catalyst: JRC-NBO-4) and niobium carbide (reference catalyst: JRC-NBO-5-C) which are expected to be utilized in optical lenses, electronic materials (piezoelectric element, capacitor, SAW filter), acidic catalysts, catalyst carriers, etc. For this reason, the structural evaluation of each sample was conducted on the basis of specific surface area, outer surface area, pore volume and pore size distribution.

Materials and Apparatus

Each of NBO-4 and NBO-5-C was processed with the following scheme, followed by particle size, pore distribution and isoelectric point measurement using the apparatus specified below.



N_2 Gas Adsorption
BELSORP MINI X
Pretreatment temperature: 200°C, 3 h in vac.
Measuring condition: N_2, 77K
BET method・t-plot method
BJH method (ca. 200 nm)

He Gas Substitution True Density Measurement
BELPYCNO
Pretreatment: He purge 10 times
Measuring condition: 10 times precision 0.02%
Measuring temperature: 25°C

Results

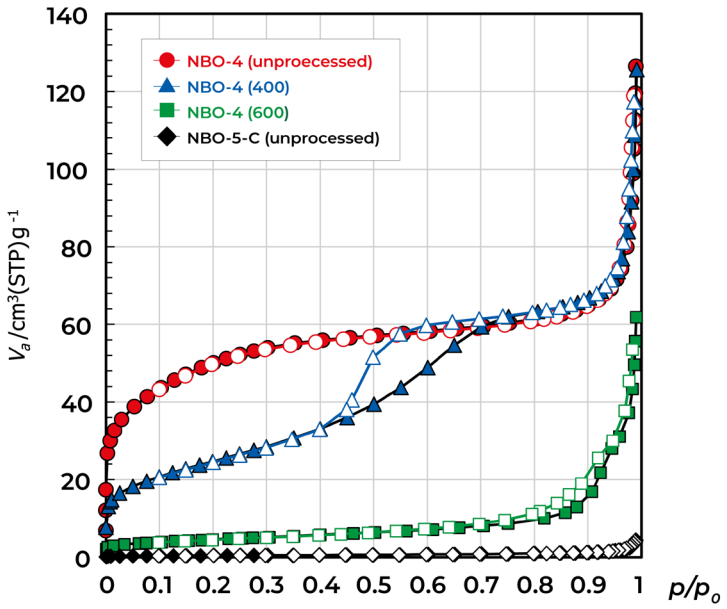


Fig. 1 Nitrogen adsorption / desorption isotherm

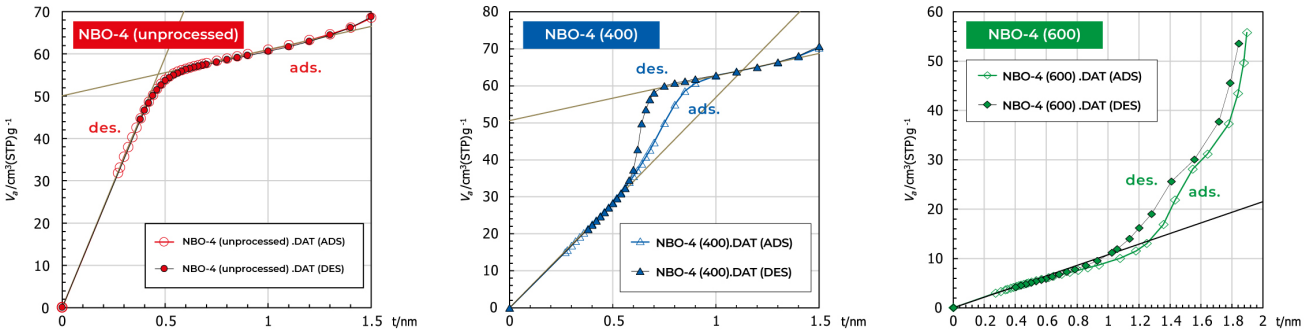


Fig. 2 t-plot (criterion: Harkins-Jura equiation)

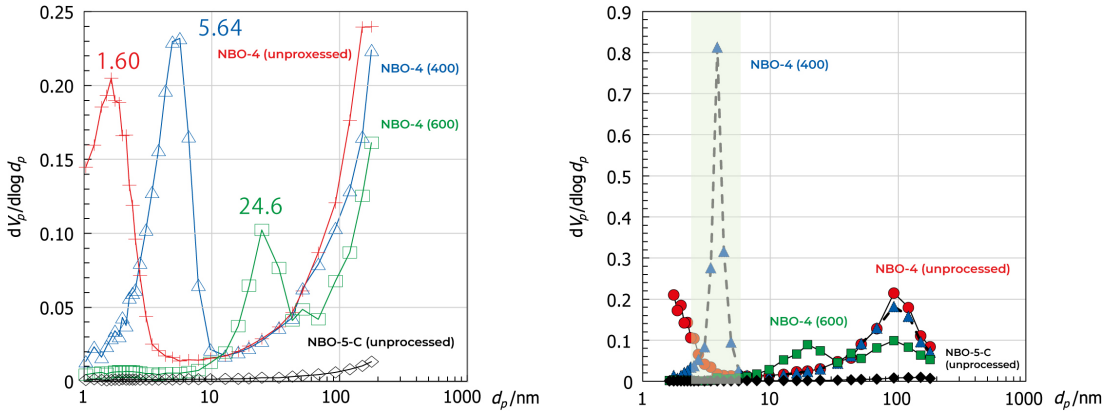


Fig. 3 BJH-plot (left: adsorbing branch, right: desorbing branch)

Table 1 Results of NBO-4 and NBO-5-C Structure Evaluation

Sample	BET method	t-Plot method (criteria: Harkins-Jura plots)				
	Specific surface area / $m^2 \cdot g^{-1}$	Total surface area / $m^2 \cdot g^{-1}$	Outer surface area / $m^2 \cdot g^{-1}$	Pore volume / $cm^3 \cdot g^{-1}$	Pore diameter (2t) / nm	True density / $g \cdot cm^{-3}$
NBO-4(unprocessed)	181	180	16.8	0.077	0.94	2.7
NBO-4 (400)	89.2	87.7	18.8	0.078	–	4.1
NBO-4 (600)	16.5	16.7	–	–	–	5.1
NBO-5-C (unprocessed)	1.71	1.68	–	–	–	7.7

Discussion

According to the results of adsorption isotherm measurement (N_2/77K), NBO-4 (unprocessed) showed a I+IV type isotherm, NBO-4 (400) and NBO-4 (600) showed a IV type isotherm, and NBO-5-C (unprocessed) showed a II type isotherm (Fig. 1). This indicates that NBO-4 (unprocessed) possesses micropores and meso-microspores, NBO-4 (400) and NBO-4 (600) possess meso-macropores, and NBO-5-C (unprocessed) is poreless. Total surface area, outer surface area and pore volume were evaluated by the BET and t-plot methods (Fig. 2) as shown in Table 1.

Evaluation of the adsorption/desprotion branches of these isotherms by t-plot revealed an outbreak of bending (typical of micropore-possessing materials) in NBO-4 (unprocessed), capillary condensation in the adsorbing branch of NBO-4 (400) and cavitation with a relative pressure of 0.42 in the desorbing branch of NBO-4 (400). These findings indicate that the pore diameter peak in the vicinity of 2 nm within the pore distribution from the desorbing branch is a so-called ghost peak (Fig. 3, dotted line). In addition, since capillary condensation of the adsorbing/desorbing branch was seen in NBO-4 (600), there seem to be irregularities on the spores. If the pore size distribution (Fig. 3) is analyzed by the BJH method (criterion t-curve Harkins-Jura), it is noticeable that the pore size peak of about 1.6 nm gradually shifts towards larger pore size as the sintering temperature gets higher. This is probably attributable to the collapse of micropores and the formation of mesopores as a result of sintering the particles that make up the NBO-4 (unprocessed). This is reflected in the reduction of specific surface area (measured by BET) and an increase in true density after increasing the sintering temperature.

Samples supplied by Reference Catalyst Panel of the Catalysis Society of Japan

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