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Niobium Powder for Electrolytic Capacitor†

1. Introduction

As the market for information and communication devices grows rapidly, so too demand is increasing for electronic components used in such devices. Capacitors are a key component of electronic devices, and the main types are the multi-layer ceramic capacitor, Al electrolytic capacitor and Ta electrolytic capacitor. The Ta electrolytic capacitor offers advantages of smaller size and larger capacitance than the others, and about 10 such capacitors are used in cell phones and about 30 in personal computers[1,2]. However, Ta is a rare metal, and the price of raw Ta ore doubled in 1980 and 2000 causing major problems, so demand for capacitors without Ta powder is increasing. Nb has almost the same chemical and physical properties as Ta, but there are larger deposits of Nb and so it is cheaper than Ta. Several attempts have been made to use Nb powder for capacitors but none has been commercialized yet. One of the reasons is that high-purity Nb powder cannot be produced by the conventional method[3]. JFE Mineral has therefore developed a high-performance Nb powder for Nb electrolytic capacitors. This paper explains the features of the developed Nb powder.

2. Production Method

The method of producing the Nb powder is outlined in **Fig. 1**. $NbCl_5$ is used as the starting material. The vapor of $NbCl_5$ is reduced by hydrogen at high temperature, then purification and sintering are performed to yield high-performance Nb powder.

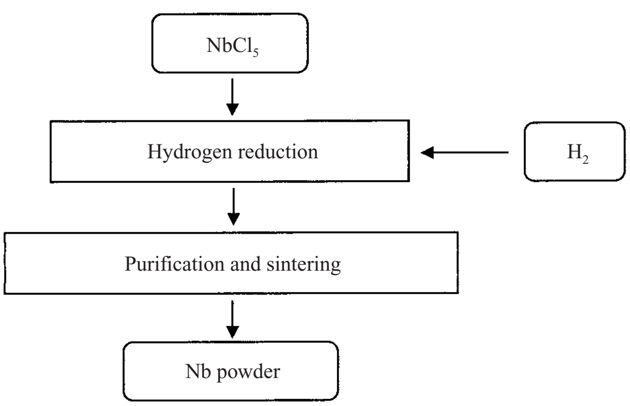

Fig. 1 Process flow for producing Nb powder

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3. Product Characteristics

3.1 Chemical Composition

The main impurities contained in Nb powder are shown in **Table 1**. The content of alkaline metals such as Na and K is less than 10 ppm and the content of transition metals such as Fe, Cr, and Ni is less than 20 ppm. Although chloride is used as a starting material, the chlorine content of Nb powder is less than 10 ppm. The oxygen content depends on the BET specific surface area of the Nb powder, and is about 8 000 to 25 000 ppm.

3.2 Powder Characteristics

3.2.1 Specific surface area and morphology of Nb powder

SEM images of three kinds of Nb powders, whose BET specific surface area is 8.0 m^2/g, 5.5 m^2/g and 3.0 m^2/g, are shown in **Photos 1** (a), (b), and (c), respectively. The primary particles of Nb powder are sintered together and form a network-like structure.

3.2.2 Particle size distribution

The particle size distribution of Nb powder whose BET surface area is 8 m^2/g is shown in **Fig. 2**. The size was measured by the laser diffraction method. If the particle size of the cumulative particle volume of x% is D_x, then D_{10} is 27 μm, D_{50} is 62 μm, and D_{90} is 109 μm.

3.3 Electrical Properties

3.3.1 Measurement method

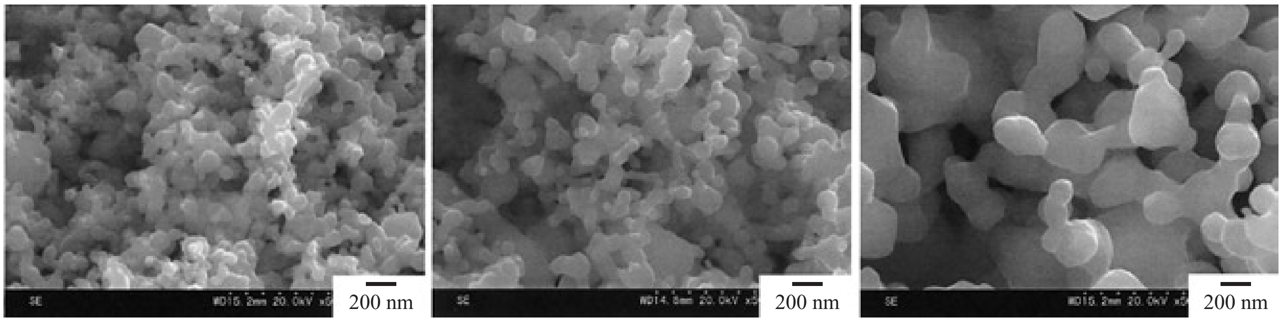
The electrical properties of Nb powder as a capacitor were measured as shown in **Fig. 3**[4]. First, an Nb pellet of cylindrical shape (diameter: 3 mm, height: 3.2 mm, density: 1.9–3.1 g/cm^3) was prepared. Nb wire as a terminal was embedded in one side of the pellet. The pellet was then sintered at 950 to 1 250°C for 30 min under reduced pressure of less than 5×10^{-3} Pa. After the sintering, the pellet was oxidized at 10 to 20 V and at 80°C for 6 h in 0.5 mass% phosphoric acid solution to form the Nb_2O_5 film as a dielectric on the surface of Nb metal. The capacitance value (CV) and leakage cur-

Table 1 Chemical impurities contained in Nb powder

(mass ppm)

Na	K	Cr	Fe	Ni	H	N	O*	C	Cl
<10	<10	<20	<20	<20	<300	<500	8 000–25 000	<150	<10

*Oxygen content depends on BET specific surface area of Nb powder.



(a) BET specific surface area = 8.0 m^2/g (b) BET specific surface area = 5.5 m^2/g (c) BET specific surface area = 3.0 m^2/g

Photo 1 SEM images of Nb powders

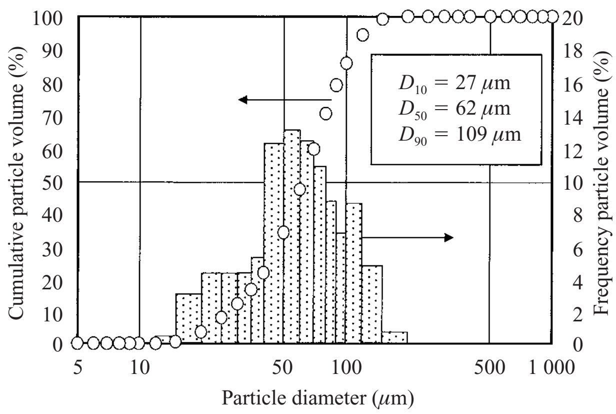


Fig. 2 Particle size distribution of Nb powder measured by laser diffraction method

rent (LC), which are important values for capacitors, were then measured in 40 mass% sulfuric acid solution. CV was measured at a DC bias voltage of 1.5 V. LC was measured at a voltage of 0.7 times the oxidation voltage, and the value at 5 min after the start of measurement was used.

3.3.2 Capacitance value

The relationship between the sintering temperature and capacitance value of three kinds of Nb powders whose BET specific surface area is 8.0 m^2/g, 5.5 m^2/g, and 3.0 m^2/g, are shown in **Fig. 4**. The CV of the Nb powder with BET surface area of 8.0 m^2/g is

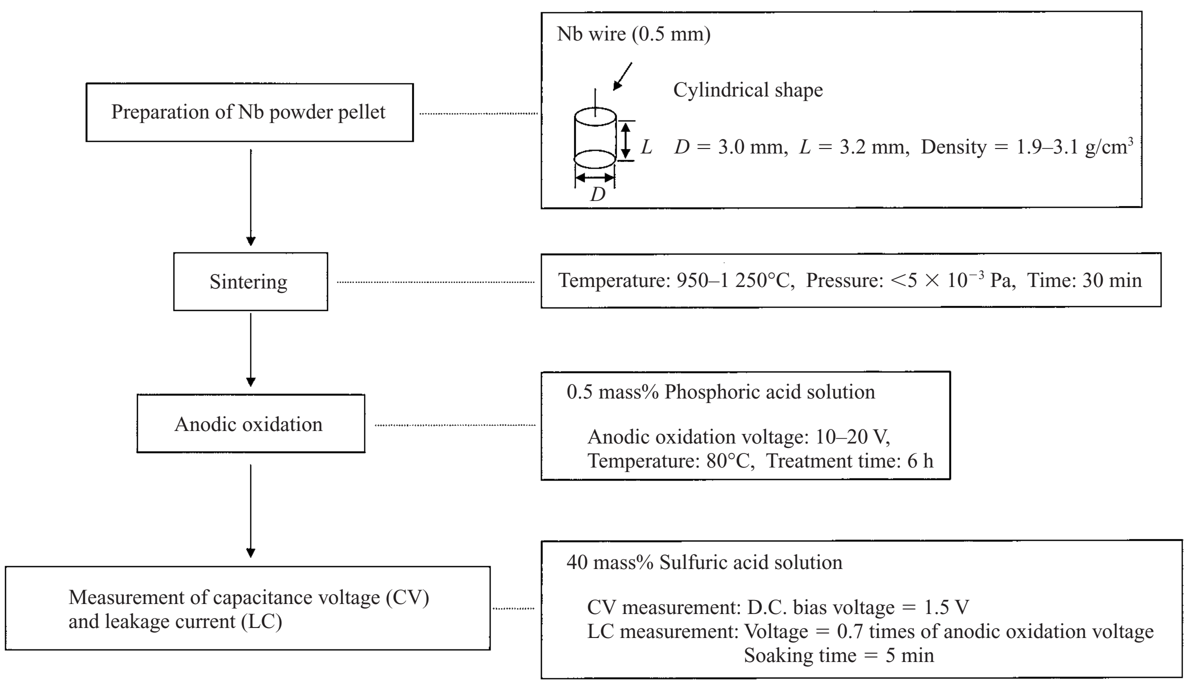


Fig. 3 Electric property measurement method of Nb powder as a capacitor

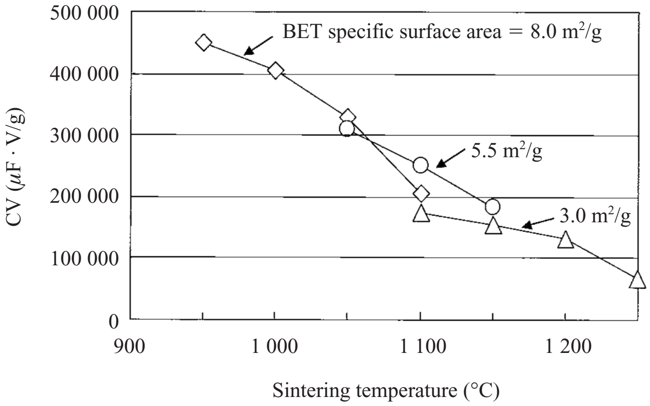


Fig. 4 Relationship between sintering temperature and CV

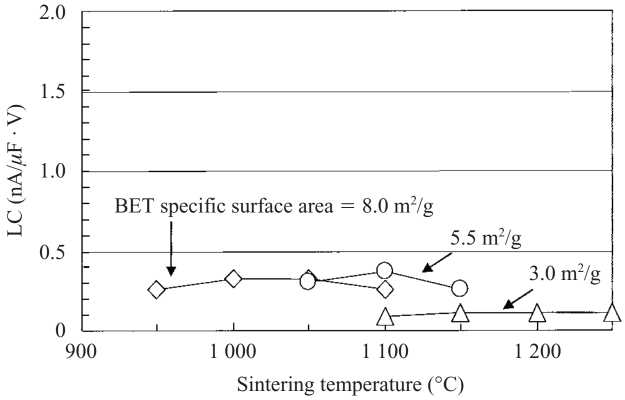


Fig. 5 Relationship between sintering temperature and LC

about 450 000 μF · V/g at the sintering temperature of 950°C; the value of the powder with BET surface area of 5.5 m^2/g is about 300 000 μF · V/g at 1 050°C; and the value of the powder with BET surface area of 3.0 m^2/g is about 180 000 μF · V/g at 1 100°C.

3.3.3 Leakage current

The relationship between the sintering temperature and leakage current of three kinds of Nb powders whose BET specific surface area is 8.0 m^2/g, 5.5 m^2/g, and 3.0 m^2/g, are shown in **Fig. 5**. The LC values of the samples are less than 0.5 nA/μF · V.

4. Conclusion

There are larger deposits of Nb than Ta, so Nb powder can be produced more cheaply and at a more stable price than Ta powder. The Nb powder developed by JFE Mineral has high purity, high BET surface area, high capacitance and low wet leakage. It is a highly promising material for compact, high-capacitance capacitors and is expected to replace the conventional materials in the near future.

References

1) Global Capacitor Market. 2003-2008, Paumanok Group, Inc.
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3) Oka, Hideo. Kinzoku. vol. 72, no. 3, 2002, p. 211.
4) EIAJ RC-2361A Test methods of tantalum sintered anodes for electrolytic capacitors. Standard of Electronic Industries Association of Japan (Feb. 2002 revised).

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