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High electrical conductivity and high corrosion resistance fibers with high modulus and high strength prepared by electroless plating of gold on the surface of poly (*p*-phenylene benzobisoxazole) (PBO)

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Abstract In an attempt to produce glittering gold fibers with high modulus and high strength, gold plating on the surface of poly(*p*-phenylene benzobisoxazole) (PBO) fibers was carried out by using an electroless plating method. Due to the difficulty in plating gold directly on organic and inorganic fibers, gold plating was carried out on the surface of copper-plated and nickel-plated fibers; for the latter the nickel was plated on the copper-plated fibers. Namely, composite fibers, termed PBO/Cu/Au and PBO/Cu/Ni/Au, were prepared. The morphology of plated fibers was studied by X-ray diffraction, scanning electron microscopy with energy dispersive spectroscopy and electrochemical polarization measurements. It was found that gold was uniformly plated on the PBO fiber, and the gold-plated fibers have good corrosion resistance. The electrical conductivities of the two kinds of gold-plated fibers were higher than 4×10^4 S/cm, and their tensile strengths and Young's moduli were greater than 1.9 GPa and 130 GPa, respectively, when estimated in terms of a single composite fiber.

Introduction

Metal coatings on fibers have been widely investigated to provide new functions to the original fibers and to develop their utilities in industrial fields. For example, the Dupont company has announced their Aracon metal clad aramid fibers yarns with the conductivity of (~0.001 ohm/cm) [1], which is promising materials for the aerospace. AmberStrand™, produced by Syscom Technology, Inc, is an electrically conductive [2], low-weight, high strength and yet flexible polymer/metal hybrid yarn. By adding a metal coating, unique engineering properties of AmberStrand™ fiber will promote numerous electronic textile applications. Furthermore, many applications about these functioned fibers have been mainly realized by Post and Orth [3–5].

To promote further developments of functional fibers, gold has been one of the most attractive materials since ancient times, and fabrics woven with gold threads have been used as high-grade materials for wall hangings, clothing and mats. The plating of gold has mainly been carried out on the surfaces of wool, silk, cotton and ramie fibers, to make embroidery floss [6]. In the electronics industry, gold plating has also been widely used to prepare the surfaces of electrical contacts and wire bonding pads of semiconductor devices, due to gold's advantageous characteristics of high conductivity, high reliability and high corrosion resistance [7–11].

Poly(*p*-phenylene benzobisoxazole) (PBO) fibers, which are well-known as Zylon, are super-heat-resistant polymers with extremely high Young's modulus values of polymers [12, 13]. PBO provides the most attractive properties and the greatest economic potential of the polybenzazoles [14–18]. In particular, because of its light weight, it is expected that it will be able to be used in many fields such as aircraft manufacture and aerospace, and a number of basic investigations into their properties have been reported [19–23]. Therefore, it is of interest to consider the production of glittering gold fibers with a high modulus and high strength by plating gold on the surface of PBO fiber. It should be

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noted here that most metals transform from the solid state to the liquid state above their melting point, and at this point their mechanical strength immediately becomes zero. On the other hand, PBO fibers start to pyrolyze at ca. 600 °C, but no sudden drastic disruption occurs, even when PBO fibers are placed in fire. Accordingly, gold-plated PBO fibers can maintain their original shape. Furthermore, gold-plated PBO fibers can recover immediately from a large deformation by bending, while in most metal wires plastic deformation takes place on bending. Such high elasticity of PBO fibers is expected to provide great potential as a new kind of material.

This paper deals with the preparation of gold-plated PBO fibers, which are glittering fibers with high modulus and high strength, by electroless plating and characteristics of the products. This technique is important not only due to the absence of the need for power supplies, but also due to the superior quality of the coating with respect to uniformity, the lower porosity, and the selective plating ability [24–28]. The objective of this research was to evaluate the effect of the electroless gold plating on the fiber morphology and the mechanical and corrosion resistant properties.

Experimental section

Sample preparation

The PBO fibers used in the present experiment had the following characteristics: filament decitex (1.7), denier (1.5), density (1.56 g/cm^3), tensile strength (5.8 GPa), Young's modulus (280 GPa), elongation at break (2.5%) and moisture regain (0.6%). They were supplied by Toyobo Co. Ltd.

It is well known that direct plating of gold on polymer fibers is difficult, since the surface of a PBO fiber is inert. Hence the copper was plated on the surface of PBO as an active layer before gold plating. Figure 1 showed the processes involved in electroless gold plating on PBO fibers.

As shown in Fig. 1, first the surfaces of PBO fibers 7 cm in length were washed with ethanol at 78 °C for 10 h, and they were then dried at room temperature. Second, a solution containing 8.0 mol/L of HCl and 1.5 mol/L of H_2SO_4 was prepared and the PBO fibers were immersed in 30 mL of this solution for 10 min at 20 °C, to produce coarse surfaces by etching. After washing, the fibers were immersed in a 30 mL solution of 2.5 mol/L for 6 min at 70 °C, as the alkali processing step. After washing, the fibers were immersed in 30 mL of 2.5 mol/L CH_3COOH for 6 min at 70 °C for neutralization, and then washed with distilled water. 4.5×10^{-3} mol/L of $PdCl_2$, 0.2 mol/L of $SnCl_2$ and 4.9 mol/L of HCl were stirred together to prepare the activation solution which had a volume of 20 mL. The fibers were stirred in the activation solution for 4 min at 20 °C. The

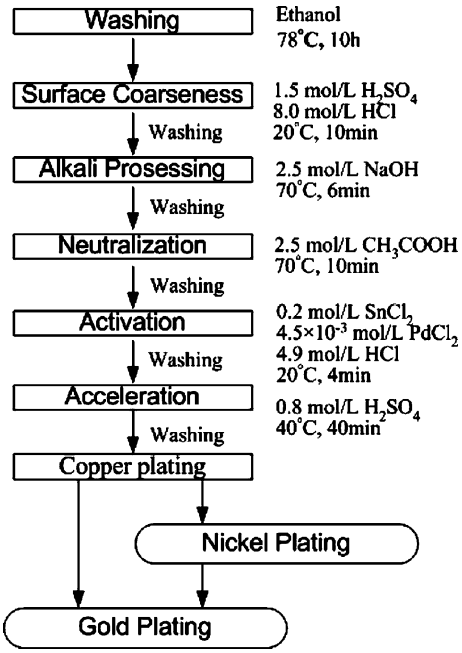

Fig. 1 Processing for electroless gold plating on PBO fibers

activated fibers were then removed from the activation medium and washed. Next, the activated fibers were stirred in 30 mL of aqueous H_2SO_4 (0.8 mol/L) for 4 min at 40 °C. After washing and drying, the PBO fibers were weighed.

The isolated fibers were put into a bath with the chemical composition listed in Table 1, to plate copper on their surfaces. Through trial and error, an immersion time of 20 min was found to be the best condition for obtaining a suitable surface to pursue gold plating. The resultant copper-plated fibers were washed, and PBO/Cu fibers were obtained.

On the basis of reports that nickel acts as a barrier layer between copper and gold to prevent unwanted intermetallic formation [29, 30], nickel was plated on the surface of some PBO/Cu fiber, and then gold was plated on the surface of the nickel. To achieve the nickel plating, the PBO/Cu fibers were activated in palladium chloride solution and plated in a nickel bath with the composition listed in

Table 1 Composition and operating condition of electroless copper plating bath

Chemical	Concentration (mol/L)
$CuSO_4 \cdot 5H_2O$	0.04
4Na-salt of ethylenediamine-tetraacetic acid (EDTA)	0.12
HCHO	0.20
Na_2SO_4	0.14
HCOONa	0.30
KCN	4.6×10^{-4}
Ph	12.5

Table 2 for 5 min. Nickel plated fibers (PBO/Cu/Ni) were obtained and then treated in the ammonium fluoride and sodium sulfonate solution. After washing with hydrazine hydrate at room temperature, the treated PBO/Cu/Ni fibers were placed in the gold baths for 20 min. The chemical components of the gold plating bath are listed in Table 3. In contrast, the PBO/Cu fibers were plated directly in the same gold bath for 20 min. The two kinds of gold-plated fibers obtained were then rinsed and dried.

Preliminary experiments confirmed that immersion of PBO fibers for 10 min in the copper plating bath, after the chemical modification shown in Fig. 1, completely changed the color of the fiber from white to yellow. This indicated that plated copper had covered the surface of the PBO fiber. The thickness of the copper increased with an increasing immersion time, but tended to level off after 60 min. When the immersion time was set at 60 min, the further increase in the diameter caused by plating nickel and gold on the copper-plated PBO fibers caused cracking and gave the plated fibers a rough surface. The immersion time that ensured the best plating of copper was fixed at 20 min as discussed above. Incidentally, the volume fraction of plated gold was almost constant for immersion times in excess of 20 min.

Table 4 shows the volume fraction (vol.%) and thickness of each plated metal layer. The calculation was carried out using the weight of plated fiber at each plating stage and the intrinsic densities of copper (8.96 g/cm^3), nickel (8.91 g/cm^3) and gold (19.3 g/cm^3). The layer thicknesses of the plated fibers were calculated by assuming that the composite fibers take a cylindrical shape. Interestingly, the gold layer plated on the nickel surface was thicker than that plated on the copper surface. On the other hand, as described previously, the thicknesses of the gold layers plated on the PBO/Cu and PBO/Cu/Ni fibers were constant when the immersion time in the gold plating bath was longer than 20 min. This indicates that the different thicknesses of gold layer reported in Table 4 are attributed to the characteristics of the substrate metals.

Table 2 Composition and operating condition of electroless nickel plating bath

Chemicals	Concentration (mol/L)
$NiSO_4$	0.1
Citric acid	0.2
$(NH_4)_2SO_4$	0.5
NaH_2PO_4	0.15
pH	7–10
Temperature (°C)	90

Table 3 Composition and operation conditions of electroless gold plating bath

Chemicals	Concentration (mol/L)
K_2CO_3	0.75
KOH	0.87
KCN	0.01
$KAu(CN)_2$	0.017
$N_2H_4{\cdot}H_2O$	0.5
Temperature (°C)	80

Table 4 Average layer thickness and volume fraction of plated fibers and PBO fibers

	PBO/Cu/Ni/Au		PBO/Cu/Au	
	Volume fraction (vol.%)	Thickness (μm)	Volume fraction (vol.%)	Thickness (μm)
PBO fiber	46	–	85	–
Copper	7	0.422	11	0.361
Nickel	35	1.781	–	–
Gold	12	0.525	4	0.126
Diameter of plated fiber	–	16.956	–	12.474

Analysis

The microstructure was observed by using a scanning electron microscope (SEM, JEOL6700) equipped with energy dispersive spectroscopy (EDS). Observation was possible on the surface but not on the cross-section, because of the difficulty in cutting and breaking the plated fibers without causing any deformation of their circular cross-section. X-ray diffraction intensity distributions were measured by a 12 kW rotating-anode X-ray generator (Rigalu RAD-rA) with monochromatic radiation (wavelength 0.1542 nm). The measurement by a reflection method was performed from 5° to 62° (twice the Bragg angle) at a scanning speed of 1°/min. A tensile tester (KES-G1, Kato Tech Co., Ltd.) was used to measure the mechanical properties of the metal-plated fibers. This is a revolutionary device developed by Kawabata to measure the tensile strength and Young's modulus of a single fiber [31, 32]. In this machine, a DC servo motor with a simple transmission mechanism was designed to minimize the mechanical noise from the tester. The motor deriving power is transmitted to the driveshaft through a helical rack-pinion mechanism similar to the driving mechanism of a microscope body tube. The electrical conductivity was measured by a four-terminal method at room temperature using an H2-3000 (Hokuto Electric Co. Ltd.), and was measured by a two-terminal method at elevated temperature (HR-100, Iwamoto Seisakusho Co. Ltd). Electrochemical measurements were carried out in a

conventional three-electrode electrochemical cell at room temperature. The working electrode was metal-plated fibers. A platinum wire was used as a counter electrode, and the reference electrode was a saturated calomel electrode (SCE). A microcomputer-based electrochemical analyzer (HZ-3000, Hokuto Denko Corporation) was employed for the electrochemical studies.

Results and discussion

Figure 2 shows photos of the three kinds of composite fibers, PBO/Cu, PBO/Cu/Au and PBO/Cu/Ni/Au. Obviously, the gold-plated fibers glitter with a golden color. The aureateness of the PBO/Cu/Au fiber is slightly better than that of the PBO/Cu/Ni/Au fiber, in spite of its thinner gold layer as listed in Table 4. However, aureate discrimination between the PBO/Cu/Au and PBO/Cu/Ni/Au fibers is very difficult in Fig. 2.

To check the plating conditions, wide-angle X-ray diffraction (WAXD) intensity distribution curves were obtained for the two kinds of gold-plated fibers. Curves (a) and (b) in Fig. 3 correspond to the different intensities from PBO/Cu/Ni/Au and PBO/Cu/Au composite fibers, respectively. Two clear diffraction peaks from the (111) and (200) planes of gold can be observed for the two composite fibers. However, the intensity curve for the PBO/Cu/Au shows the diffraction peaks from the (010) and (210) planes of PBO crystallites in addition to the peaks from the (002), (111) and (200) planes of copper. These peaks are attributed to very thin gold layer and thin copper layer, so that incident X-ray beams pass through the two layers. In contrast, the PBO/Cu/Ni/Au composite fiber provided only a weak peak from the (002) plane of copper and an overlapped peak from the (200) plane of gold and the (111) plane of nickel, but no peak from PBO crystallites. However, the peaks from the (200) plane of gold in curves (a) and (b) have similar heights. Accordingly, the peak contribution from gold is thought to be greater than that from nickel. This means that the thick gold layer completely covers the surface of the PBO/Cu/Ni fibers, and that the layer is thick enough to prevent penetration of X-ray beams through the gold layer. This supports the results in Table 4.

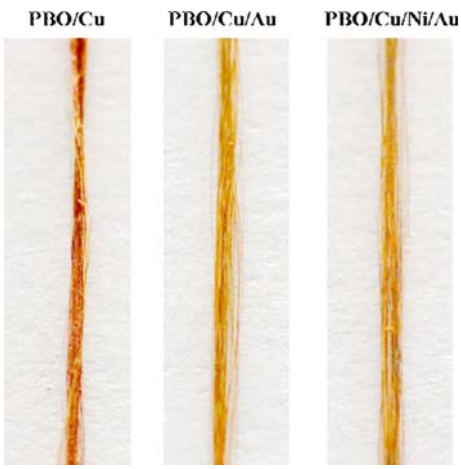

Fig. 2 Photos of metal-plated PBO fibers

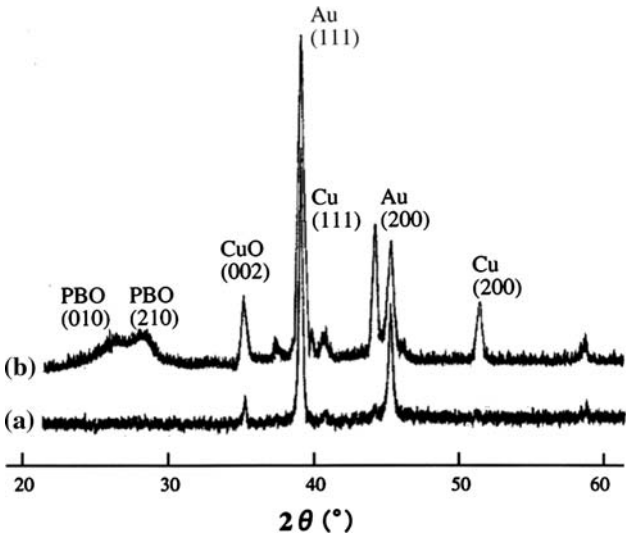

Fig. 3 X-ray diffraction intensity distribution of (**a**) PBO/Cu/Ni/Au and (**b**) PBO/Cu/Au

Figure 4 shows SEM images of the gold-plated fibers. Many gold particles cover the surfaces of the two kinds of fiber. However, detailed observation reveals that the average size of gold particles on PBO/Cu/Ni/Au composite fiber is smaller than that on the surface of PBO/Cu/Au fiber, and that the former surface is smoother than the latter surface, indicating that plating of small gold particles on the nickel layer is more uniform than that on the copper layer. At the same magnitude, the apparent diameter of PBO/Cu/Ni/Au composite fiber is obviously thicker than that of PBO/Cu/Au because of the addition of a nickel layer between the copper and gold layers. Each diameter is listed in Table 5 as an average of the diameters of ten plated fibers measured by a ruler in the present SEM.

The average diameters of PBO/Cu/Au and PBO/Cu/Ni/Au fibers in Table 5, estimated by direct observation, are slightly larger than those in Table 4, estimated from volume fractions. The values in Table 5 contain voids between adjacent metal particles, while the values in Table 4 were calculated by assuming uniform metal layers. As listed in Table 5, the gold layer of the PBO/Cu/Ni/Au fibers was confirmed to be thicker than that of the PBO/Cu/Au fibers.

Figure 5 shows EDS photos of mapping analysis, which are shown to allow analysis of the element distribution. In these photos, blue dots indicate the presence of copper

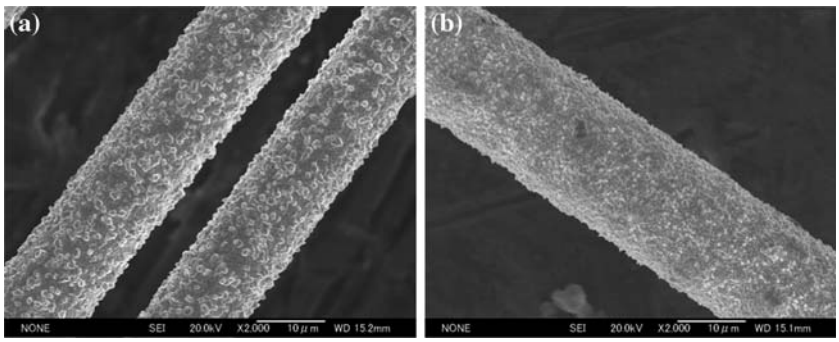

Fig. 4 SEM images of metal plated PBO fibers: (**a**) PBO/Cu/Au; (**b**) PBO/Cu/Ni/Au

Table 5 Average diameters of metal plated PBO fibers

Sample	PBO	PBO/Cu	PBO/Cu/Au	PBO/Cu/Ni/Au
Diameter (μm)	11.500	12.552	13.188	17.156

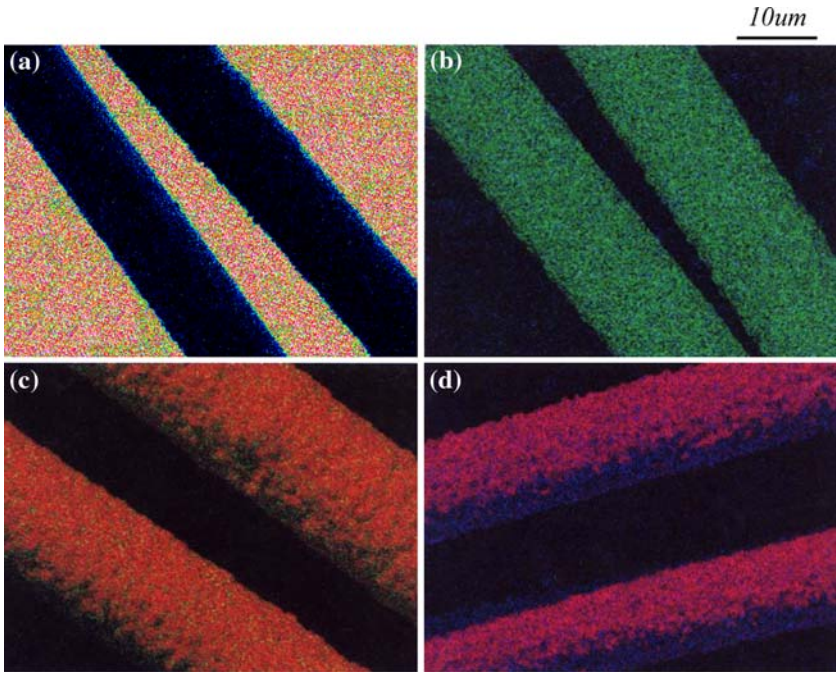

Fig. 5 EDS mapping analysis for metal plated PBO fibers: (**a**) PBO/Cu, (**b**) PBO/Cu/Ni, (**c**) PBO/Cu/Ni/Au, (**d**) PBO/Cu/Au

element; green dots (photos (b) and (c)), nickel element and red dots (photos (c) and (d)), gold element. In photos (c) and (d), it can be seen that the composite fibers are covered by gold particles. In photo (d), the blue color on the surface of the fiber indicates that the copper-plated layer was not completely covered by gold particles. In photo (c), the green color appears in some places, but the area of green is less than that of blue in photo (d). This means that nickel plays a significant role as an active layer for gold plating. Here, as shown in photo (c), it may be expected that the nickel particles can be seen due to imperfect gold plating. However, there were no X-ray diffraction peaks from nickel particles in Fig. 3. This indicates that most of the surface was covered by a thick gold layer.

Figure 6 shows typical examples of stress versus strain measurements for the PBO/Cu, PBO/Cu/Au and PBO/Cu/Ni/Au fibers. Each result was obtained as the average of the measurements for 10 fibers, to ensure reproducibility. The volume fractions of PBO/Cu/Au and PBO/Cu/Ni/Au fibers are listed in Table 4 and the volume fraction of Cu is 7%. The Young's modulus and tensile strength values obtained from the stress–strain curves are listed in Table 6. The Young's modulus and tensile strength of the PBO/Cu fibers

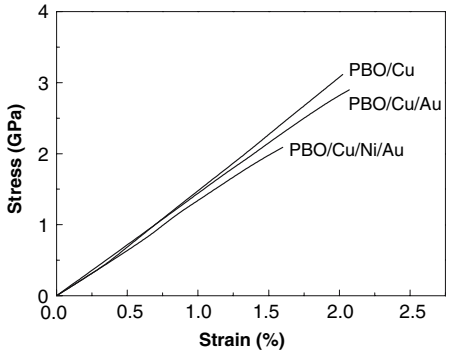

Fig. 6 Stress–strain curves for metal-plated PBO fibers

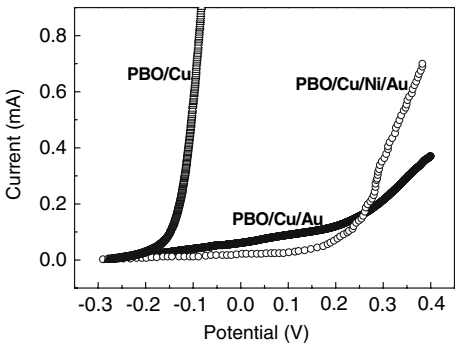

Fig. 7 Anodic polarization of metal-plated PBO fibers in 3% NaCl solution

Table 6 Electrical and mechanical properties of plated and non-plated PBO fibers

Sample	Electrical conductivity (S/cm)	Young's modulus (GPa)	Tensile strength (GPa)
PBO/Cu	3.56×10^4	174	3.1
PBO/Cu/Au	4.20×10^4	140	2.7
PBO/Cu/Ni/Au	4.67×10^4	130	1.9

are higher than those of the PBO/Cu/Au and PBO/Cu/Ni/Au fibers. These values decreased with increasing thickness of the plated layer, since the intrinsic Young's moduli and tensile strengths of copper, nickel and gold are lower than the Young's modulus (280 GPa) and tensile strength (5.8 GPa) reported for a PBO fiber.

The conductivity of metal-plated fiber increased drastically from $10^{-12.8}$ S/cm, corresponding to the conductivity of PBO, to 10^4 S/cm. The conductivity became higher as the thickness of the plating layers increased. This is reasonable, since PBO is an insulator.

The corrosion behavior of gold-plated fibers was also investigated in a chloride solution. Before electrochemical measurements, the gold-plated fibers were immersed in a 3% NaCl solution with a pH of 7.0 for 24 h. Figure 7 shows the anodic polarization curves of the plated fibers. The breakdown potential, at which the current starts to flow freely, shifted to a positive value for the gold-plated fiber. This means that the gold layer provided corrosion resistance. Of the two kinds of gold-plated fiber, the PBO/Cu/Ni/Au fiber is slightly more stable than the PBO/Cu/Au fiber, since the current for the PBO/Cu/Ni/Au fiber stays almost at zero up to 0.1 volt, while the current for the PBO/Cu/Au fiber increases slowly with increasing potential. This difference is due to the thicker gold layer of the PBO/Cu/Ni/Au fiber. In any case, it is evident that the combined nickel/gold metallization provided excellent corrosion resistance.

Figure 8 shows SEM images of the plated fibers after the electrochemical measurements. In Fig. 8(a), plated copper particles disappear, since the plated copper was dissolved in the solution. Hence the observed SEM image is not clear because of the decreased electrical conductivity of the residual fiber. However, under the same SEM observation conditions, it is seen that most of the gold particles remained on the surfaces of the fibers in images (b) and (c), although there are some corrosion pits. This indicates that the gold-plating improved the corrosion resistance of the plated fibers.

Figure 9 shows the temperature dependence of the electrical conductivity over the temperature range from room temperature to 200 °C. The conductivities of PBO/Cu/Ni/Au and PBO/Cu/Au fibers are 4.67×10^4 S/cm and 4.20×10^4 S/cm at room temperature indicating the very small difference within the experimental error, although the metal layer thicknesses of the both fibers are quite different. The conductivities are almost constant over the given temperature range. No peeling of metals by thermal expansion was confirmed during the measurements. The temperature dependence of the plated fibers is reasonable, since the conductivities of plated metals are hardly affected by the indicated temperatures.

To check the reason, the electrical conductivities $\sigma_1$and σ_2 of the composite fibers of PBO/Cu/Ni/Au and PBO/Cu/Au respectively, are calculated by the following equation:

$$\sigma_1 = \sigma_{Cu}\varphi_{Cu} + \sigma_{Ni}\varphi_{Ni} + \sigma_{Au}\varphi_{Au} + \sigma_{PBO}\varphi_{PBO} \quad (1)$$

where

$$\varphi_{Cu} + \varphi_{Ni} + \varphi_{Au} + \varphi_{PBO} = 1 \quad (2)$$

and

$$\sigma_2 = \sigma_{Cu}\varphi_{Cu} + \sigma_{Au}\varphi_{Au} + \sigma_{PBO}\varphi_{PBO} \quad (3)$$

where

$$\varphi_{Cu} + \varphi_{Au} + \varphi_{PBO} = 1 \quad (4)$$

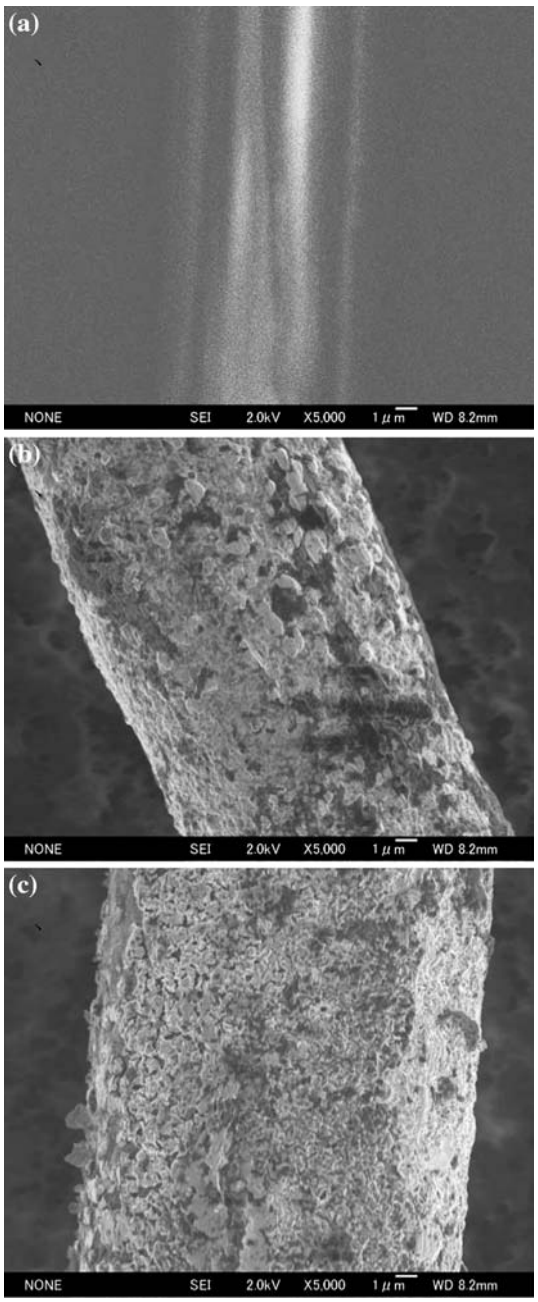

Fig. 8 SEM images of corroded fibers: (**a**) PBO/Cu; (**b**) PBO/Cu/Au; (**c**) PBO/Cu/Ni/Au

In Eqs. (1) and (3), σ_{Cu}, σ_{Ni}, σ_{Au} and σ_{PBO} are electrical conductivity φ_{Cu}, φ_{Ni}, φ_{Au} and φ_{PBO} are the volume fraction of Cu, Ni, Au and PBO, respectively. Here the electrical conductivity of σ_{PBO} is very small in comparison of other conductivities of metals, and the term of PBO can be neglected. The electrical conductivities of PBO/Cu/Ni/Au and PBO/Cu/Au fibers, calculated by Eqs. (1) and (3), are 1.46×10^5 S/cm and 8.36×10^4 S/cm, respectively.

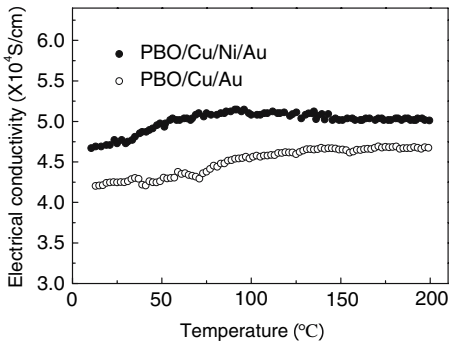

Fig. 9 Temperature dependence of the electrical conductivity of metal-plated PBO fibers

The calculated values support the small difference between the conductivities of the PBO/Cu/Ni/Au and PBO/Cu/Au fibers in Fig. 9.

Conclusion

Gold-plated PBO fibers were successfully prepared as PBO/Cu/Au and PBO/Cu/Ni/Au composites, using copper and nickel layers. The volume fraction of plated gold particles was saturated after 20 min of immersion in the gold-plating bath. The electrical conductivity of gold-plated fiber was greater than 10^4 S/cm, although the conductivity of PBO fiber was $10^{-12.8}$ S/cm. The conductivity of PBO/Cu/Ni/Au fiber was higher than that of PBO/Cu/Au fiber because of an increase in the thickness of plated layers due to the nickel plating. In contrast, the average Young's modulus and tensile strength were 130 and 1.9 GPa, respectively, for the PBO/Cu/Ni/Au fibers, and 140 and 2.7 GPa, respectively, for the PBO/Cu/Au fibers. This is because the Young's moduli and tensile strengths of copper, nickel and gold are lower than those of PBO fibers. The corrosion resistance was improved greatly by gold plating on the copper- or nickel-plated PBO fibers. The gold layers plated on the nickel layers were thicker than those plated on the copper layers. However, the glittering gold color of the PBO/Cu/Au fibers was more beautiful than that of the PBO/Cu/Ni/Au fibers, indicating that the glittering gold color depends on the characteristics of the different plated metals. From our experimental results, it may be concluded that the PBO/Cu/Au and PBO/Cu/Ni/Au fibers can be used both as industrial fibers and as gold threads for weaving high-grade fabrics. Furthermore, it may be expected that the gold-plating fibers with attractive mechanical, thermal and corrosion resistant properties can be applied to decorative wall hangings and mats.

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