Fabrication of a Graded-Index Polymer Optical Fiber Preform without Cavity via the Automatic Refilling Process

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ABSTRACT: When two monomers with different densities and refractive indices are polymerized under a centrifugal force field, a cavity is generated in the rotational axis as a result of inherent volume shrinkage. Accordingly, an additional monomer-refilling process is necessary to compensate for the undesirable cavity. In this study, we modified the stepwise refilling process to an automatic process and have successfully fabricated a graded-index polymer optical fiber preform without a cavity. The process could also reduce the processing time and enhance the transmission speed of a polymer optical fiber compared with the stepwise process. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 99: 2395–2400, 2006

Key words: polymer optical fibers; refractive index; centrifugal force field; volume shrinkage; automatic refilling process

INTRODUCTION

No matter what types of information is involved (data, voice, or images), the quantities of data to be transmitted are constantly increasing in every field. Fiber optic technology, which is already widely used in telecommunication, is now emerging also as a cost-efficient and secure transmission medium over short distant within or between buildings.[1]

Up to now, step-index glass optical fibers (SI-GOFs) have been used for long distant data communications because of their inherently low attenuation and high bandwidth. Although SI-GOFs have many advantages, the limitation of the core diameter makes them ineffective in situations that require frequent connections between fibers. Consequently, copper cables have been used for short distance-data transmission media in local area networks (LANs). With the abrupt increase of data communication, the copper cables, unfortunately, were unable to cope with these capacities any more. As a candidate for solving these problems, graded-index polymer optical fibers (GI-POFs) were introduced as new transmission media, also usable for lighting and sensors. Such uses are possible because GI-POFs can have a core with large diameter in company with their high flexibility, enabling a smaller bending radius than silica-based optical fibers. One of the largest advantages of the POF should be an excellent mechanical flexibility even if it has a large diameter, such as several hundred micrometers to 1 mm, compared with the silica optical fibers.[2,3] The large core diameter enables fibers to be easily connected to each other, and thus can reduce the installation cost.

Generally, GI-POFs can be fabricated by the interfacial gel-polymerization[4–6] and ultracentrifugal force method[7,8] proposed by Koike and van Duijnhoven, respectively. The interfacial gel-polymerization technique exploits the exclusive diffusion of bulky dopants (nonpolymerizable species) with highly refractive indices, and the ultracentrifugal force method utilizes a density gradient of two materials: one has relatively lower density and a higher refractive index, and the other has higher density and a lower refractive index, generated by centrifugal force. The interfacial gel-polymerization method reveals some shortcomings of poor long-term stability caused by the dopants migration and the limitation of preform size.[9] Considering these points, it seems more desirable to form a GI profile using a gradient of copolymer composition, which is available in the ultracentrifugal force method. The ultracentrifugal force method can generate a GI profile through a gradient of copolymer composition, but has critical shortcomings of central cavity being created inevitably by volume shrinkage.[10,11] In our previous study, we reported that a cavity could be filled by the stepwise inclusion of additional monomers, but in this study, we changed the stepwise

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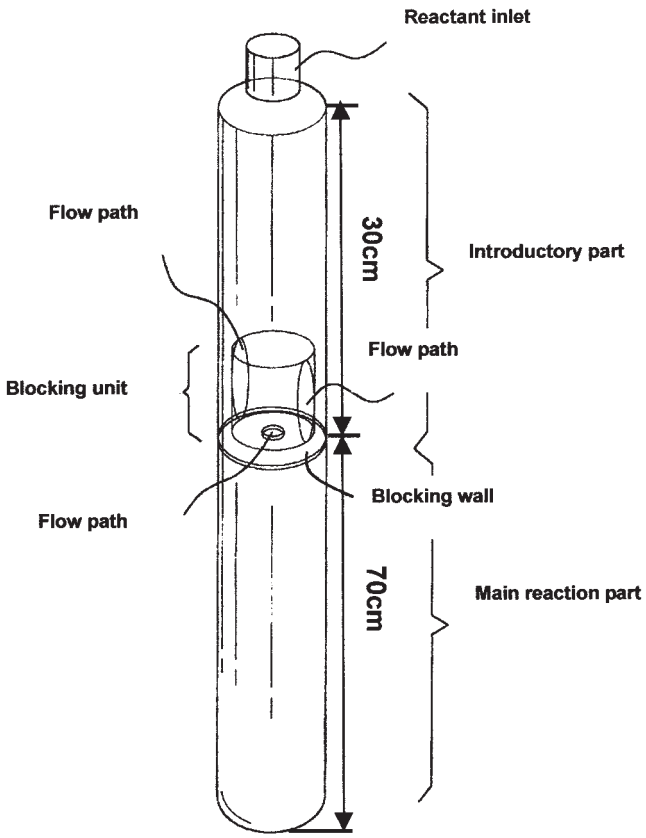

Figure 1 Schematic illustration of the modified reactor, refilling additional monomers automatically.

process into an automatic process. The modified process required less processing time and enhanced data-transmission speed of an optical fiber compared with the stepwise process.

EXPERIMENTAL

Materials

We purchased the following products from Aldrich: methyl methacrylate (MMA), benzyl methacrylate (BzMA), α,α'-azobisisobutyronitrile (AIBN) as an initiator, and *n*-butyl mercaptan (*n*-BM) as a chain transfer agent. We purified the monomers MMA and BzMA by distillation under reduced pressure after extracting inhibitors with an aqueous 0.5*N* sodium hydroxide solution, washing with distilled water, and drying with sodium sulfate.

Reactor design

A whole reactor is described elsewhere in detail.[12,13] A reactor is schematically depicted in Figure 1. It is composed of an introductory and a main reaction parts. The introductory part plays an important role in compensating for the generated cavity in main reaction part for a reactant (monomer mixture). The reactant is inserted into the reactor through a reactant inlet and also flows into the main reaction part through a blocking unit flow path. A blocking wall prevents the reactant in the main reactor part from moving to the introductory part by centrifugal force. The main reaction part is where the reactant is polymerized and a final preform without cavity is obtained. The inner diameter and the length of the introductory and main reaction parts used in the present study are 5.5 and 30 cm, and 5.5 and 70 cm, respectively.

RESULTS AND DISCUSSION

The fabrication procedure of a cavity-free GI-POF preform was schematically illustrated in Figure 2(a). First, we fabricated a cladding of 0.9 cm in thickness by polymerizing MMA of 1150 mL, containing 0.03 mol % of AIBN and 0.3 wt % of *n*-BM as a thermal initiator and a chain transfer agent, respectively, at a revolution speed of 5000 rpm and a temperature of 75°C for 24 h. As illustrated in Figure 2, the cladding was formed only at the main reaction part, because the blocking wall prevented MMA from moving to the introductory part by centrifugal force. An initial thickness of the cladding formed by MMA was 1.1 cm, but the final thickness of the cladding was 0.9 cm because of volume shrinkage caused by the density difference between MMA (density = 0.936 g/cm^3) and PMMA (density = 1.19 g/cm^3), during polymerization.

Next, we completed a GI-POF preform by fabricating a core part. After cooling the reactor to room temperature, MMA prepolymer-BzMA mixture (MMA prepolymer/BzMA = 85/15 mol %/mol %, MMA prepolymer was prepared by quenching bulk-radical polymerization of MMA when its conversion reached 0.3) of 750 mL was filled into the main reaction part. In the present study, MMA prepolymer was used so as to not to disturb the GI profile because the density of MMA is lower than BzMA (density = 1.04 g/cm^3), but the MMA prepolymer has a larger density. A MMA-BzMA mixture (MMA/BzMA = 80/20 mol %/mol %) of 700 mL was then filled into the introductory part to make the core part. Both mixtures also contained 0.03 mol % of AIBN and 0.3 wt % of *n*-BM. MMA prepolymer-BzMA and MMA-BzMA mixtures were also filled in the main reaction and introductory parts, respectively, to generate polymerization rate-difference between both parts.

If the polymerization rate of the reactants in both parts is the same, the reactant in the introductory part cannot compensate for the cavity generated in the main reaction part because both reactants solidify at the same rate, and thus the reactant in the introductory part chocks the flow paths and cannot flow into

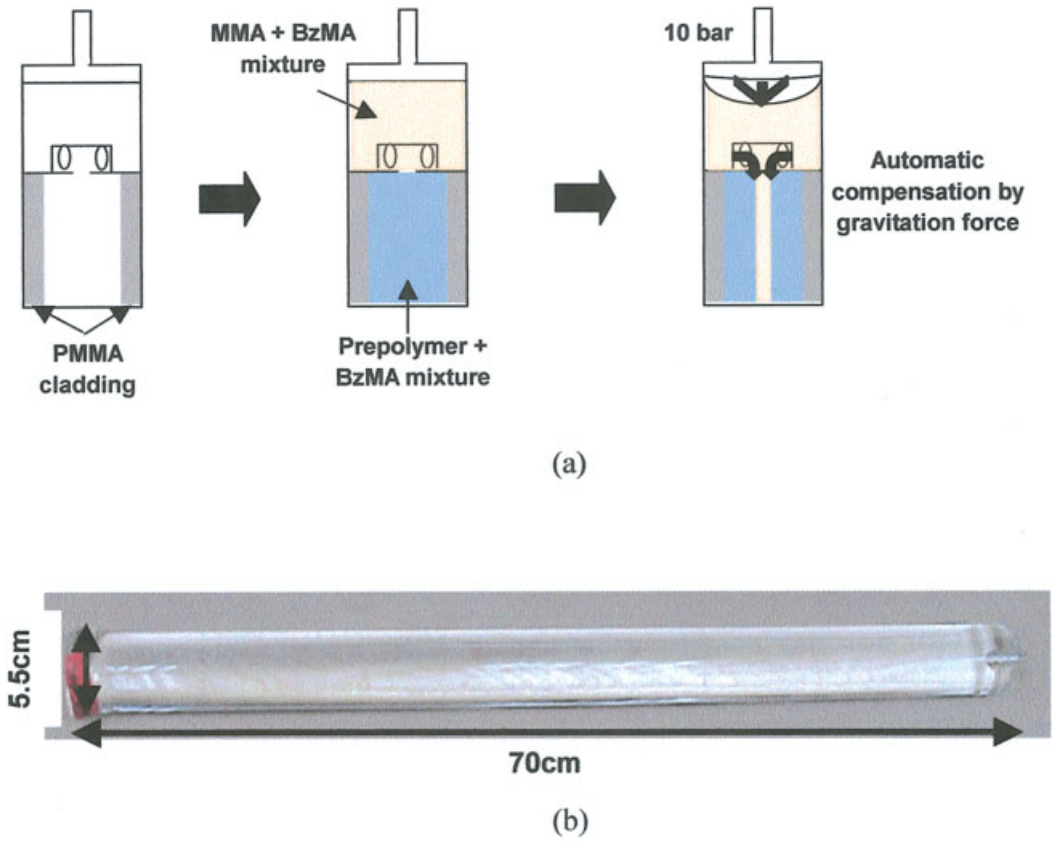

Figure 2 (a) Processing procedure and (b) photograph of a GI-POF preform without cavity obtained by the process. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

the main reaction part. Actually, a preform with a cavity is obtained when the polymerization rate in both parts is similar. The polymerization rate difference is caused by the Trommsdorff effect,[14] that is, the MMA prepolymer-BzMA mixture in the main reaction part having higher viscosity than that of the MMA-BzMA mixture in the introductory part is polymerized more quickly.

Temperature was then elevated up to 75°C at a rate of 5°C/min, rotating the reactor at 3000 rpm to introduce polymerization. As the temperature reached 75°C, the revolution speed was gradually reduced from 3000 to 1000 rpm, at a rate of 100 rpm/h to compensate for the cavity generated in the main reaction part with the polymerizing monomer mixture in the introductory part. The polymerizing mixture in the introductory part is automatically filled into the cavity by gravitational force. During the polymerization, we also pressurized the introductory part with inert gas (nitrogen) up to 10 bar to enhance the force pushing the polymerizing mixture into the cavity. The obtained GI-POF preform was shown in Figure 2(b), clearly indicating that central cavity can be compensated by the automatic refilling process. Finally, a GI-POF was obtained by thermally drawing the preform.

The refractive index of the drawn fiber was measured by microscopic interferometry,[15] as schematically illustrated in Figure 3(a), and its interference-fringe pattern was shown in Figure 3(b). The fiber was immersed in matching oil whose refractive index was nearly equal to the cladding of the fiber, and thus we can consider that the ray passes through the fiber almost directly. Accordingly, we can estimate the refractive index by computing the phase shift in the fiber. The parabolic shape of the interference-fringe pattern shows that the shape of the refractive index profile is also parabolic. The fringe pattern clearly shows that the refractive index profile is smooth and continuous. The computed refractive index from the fringe pattern is shown in Figure 3(c).

The GI profile of the fiber in the core region is usually approximately determined by the power law equation[16–21]:

$$n(r) = n_1\left[1 - 2\Delta\left(\frac{r}{a}\right)^g\right]^{1/2} \tag{1}$$

$$\Delta = \frac{n_1^2 - n_2^2}{2n_1^2} \approx \frac{n_1 - n_2}{n_1} \tag{2}$$

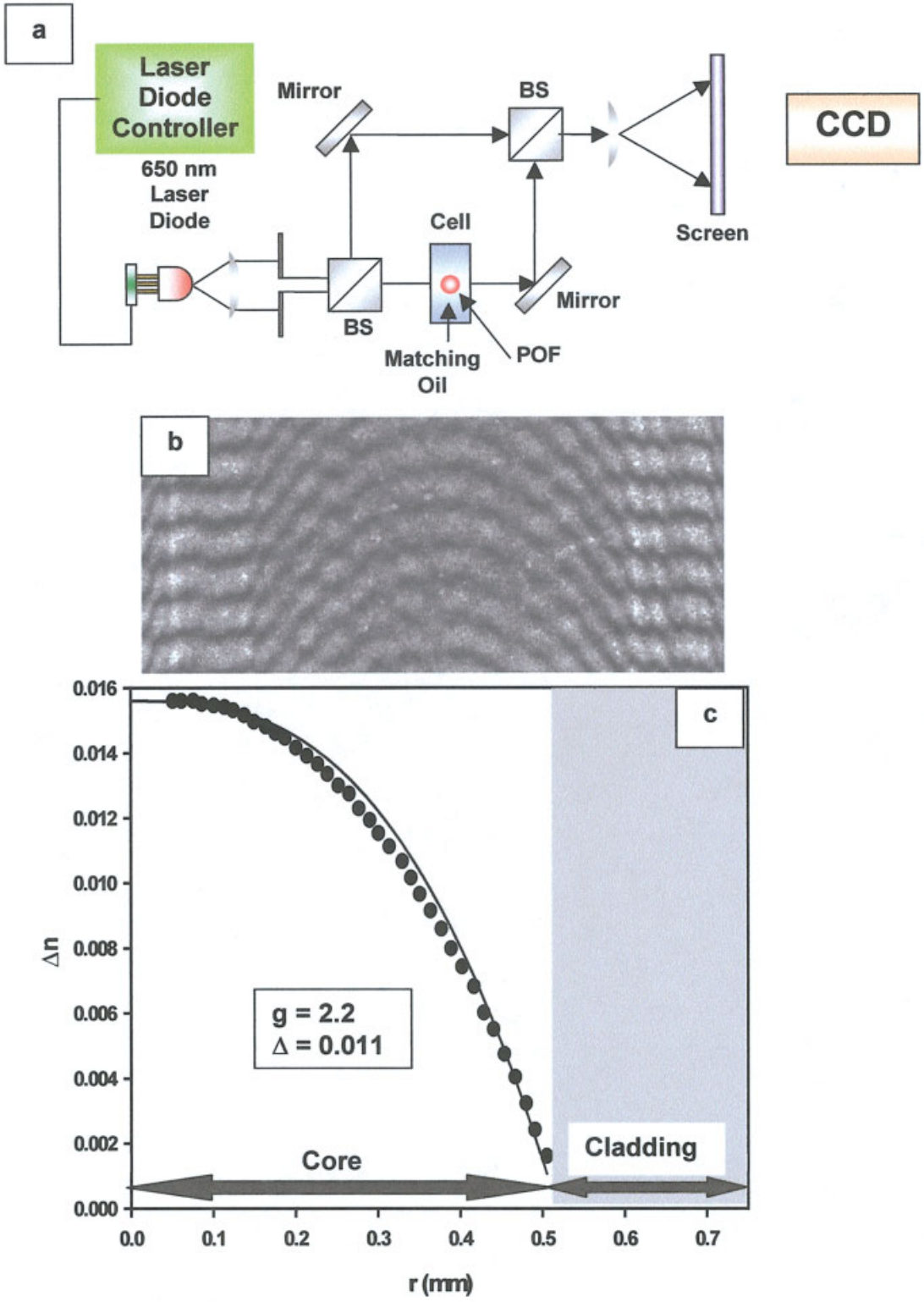

Figure 3 (a) Illustration of microscopic interferometry setup, (b) photograph of the interference-fringe pattern of the GI-POF, and (c) refractive index profile with a radial distance; dotted line indicates measured values and solid line indicates evaluated values. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

where n_1 and n_2 are the refractive indices of the core center and the cladding, respectively, a the core radius, and g the index exponent. The Δ and g values are very important because they are directly related to the transmission speed. The transmission speed is highest when g is about 2 and Δ is 0.01–0.02.[16–21] To evaluate

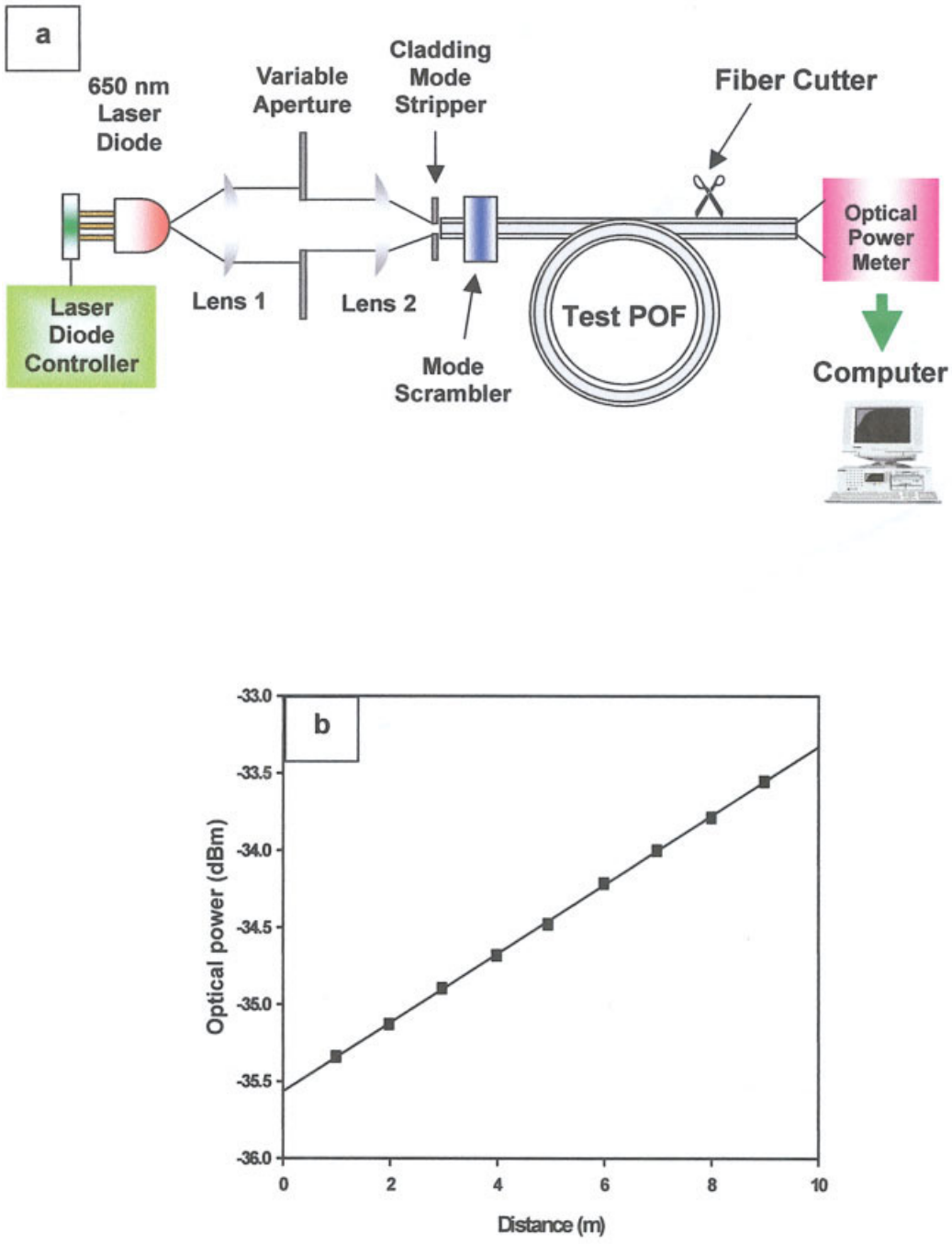

Figure 4 (a) Schematic illustration of a cutback method and (b) attenuation evaluated from it. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Δ and g of the core part, we plotted $\log\{1-[n(r)^2/n_1^2]\}$ with $\log(r/a)$. In Figure 3(c), the slope corresponds to g and the extrapolated value at $\log(r/a) = 0$ corresponds to $\log 2\Delta$.[10,11] The evaluated g and Δ are 2.2 and 0.011, respectively.

The attenuation and bandwidth of the fiber were measured by the cutback method [see Fig. 4(a)] and pulse broadening method,[16–22] and their values were 198 dB/km (light source = 650 nm) and 2.6 Gbps at 100 m, respectively. Attenuation was estimated from the slope of the graph [see Fig. 4(b)]. These attenuation and bandwidth are sufficient values for short distance-optical communications.

CONCLUSIONS

We could successfully fabricate a GI-POF preform without a central cavity through the modified automatic-refilling process. The thermally drawn GI-POF showed sufficient attenuation (198 dB/km) and band-

width (2.6 Gbps at 100 m) satisfying IEEE1394b for short distance-optical communications. Therefore, the obtained GI-POF can be used for LANs, small home and office networks, and image guides.

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