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Development of polycrystalline yttrium aluminum garnet (YAG) fibers

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ABSTRACT

Polycrystalline yttrium–aluminum garnet (YAG) fibers are attractive for high-power lasers and for high-temperature structural materials. Processing methods for <30 μm diameter polycrystalline YAG fibers suitable for single-mode laser operation are presented. The methods use extrusion of classified YAG powders with binders. Extrusion rheologies and rates, and heat-treatment temperatures, times, and environments that yield the most dense, defect-free fibers were explored. Fiber tensile testing, followed by fractography, was used to identify defects and to guide determination of optimal processing conditions. The effects of processing variables on fiber microstructures and properties are discussed. Advantages and disadvantages of processing methods are compared.

1. Introduction

Fiber lasers can be smaller, lighter, and more powerful than slab lasers [1–3]. They eliminate free-space optics, which facilitates development of more robust and reliable optical systems. Fiber lasers currently use silica cores and can achieve high power in broad bandwidth use, but are power limited for the single-mode operation necessary for beam propagation over long distances [4]. Low silica thermal conductivity causes high thermal gradients during laser operation at high power, which in turn causes mechanical failure and thermal lensing that degrades beam quality. Longer fibers ameliorate these problems, but in single-mode operation create other problems from stimulated Raman scattering (SRS) and stimulated Brillouin scattering (SBS). Yttrium aluminum garnet ($Y_3Al_5O_{12}$, YAG) has much higher thermal conductivity than silica, ~11 W/m K vs 1.37 W/m K for SiO_2 [3,5–9]. This enables higher fiber power generation without wavefront distortion or catastrophic failure from local hot spots. YAG also has a higher laser damage threshold than silica [10,11], does not photodarken as silica does [12], and has a peak SBS gain coefficient at least two orders of magnitude lower than silica [13–16]. The shorter fiber lengths possible with YAG would further reduce SBS.

The relative performance of YAG and silica fibers can be estimated [17,18]. For silica fibers in single-mode operation, there are three power limiting mechanisms: SBS, thermal lensing, and pump limitations. The maximum power is 0.2 kW for a 30 μm diameter fiber, which is the maximum diameter possible for single-mode operation. Predicted maximum power levels in YAG are 1.1 kW for a 30 μm diameter fiber, but may be as high as 20–50 kW if SBS gain coefficients in YAG are 10^{-12} m/W [4,13,15–17]. SBS and pump limitations are the only power limiting mechanisms. The high thermal conductivity and small change in refractive index with temperature of YAG eliminate thermal lensing as a power-limiting mechanism for small diameter fibers. Improvements in laser pumping will result in even higher power for YAG fiber diameters and short fiber lengths.

Melt-grown single-crystal YAG slabs are used in high-power lasers [19], and single-crystal YAG fibers have been demonstrated for both laser and structural applications [20–26]. Fiber diameters were typically limited to >75 μm, which does not allow single-mode operation, but recently single-crystal fibers with diameters as small as 20 μm have been grown by laser heated pedestal growth [15,16]. Super-heated YAG melt separates into two immiscible liquids that crystallize a $YAlO_3$–Al_2O_3 eutectic instead of YAG [27–31], so the melt must be held in a narrow temperature window just above the melting point, which makes growth of single-crystal YAG unusually difficult. Optical clarity is often inadequate because of porosity, and distribution of some dopants, such as Nd, can be non-uniform from zone refinement during

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growth [32]. Dopant inhomogeneity and production cost and reproducibility are concerns for high-power fiber lasers of single-crystal YAG.

Polycrystalline (ceramic) YAG slabs are an efficient laser host [33–36]. Laser output power over 100 kW has recently been demonstrated in YAG slabs [36]. It has higher fracture toughness than single-crystals and supports higher power densities. Two methods are used to achieve optical quality YAG slabs. One uses stoichiometric YAG powder, and the other uses Al_2O_3 and Y_2O_3 powder mixtures that react to YAG. However, to date all high power YAG slab lasers have been made using Konoshima YAG powder [36]. The processing methods are adaptable to larger and more complex shapes than single-crystals. However, the densification of polycrystalline YAG is inhibited by low diffusion coefficients [37–41]. Densification temperatures >1700 °C form grains >3 μm [34,42–44], but this may be acceptable for 30 μm diameter fiber lasers if optical quality is preserved.

Another possible application of polycrystalline YAG fiber is fiber reinforcements in ceramic matrix composites. Currently non-oxide and oxide fibers are commercially available [45,46]. Due to high oxidation resistance, oxide fibers can be used in oxidation environment but its low creep resistance at high temperature could be problematic. Among oxides, YAG is one of the most creep and corrosion resistant oxides [47], and consequently is also of interest as a high-temperature structural ceramic. Several researchers produced small amounts of polycrystalline YAG fiber for structural applications [48–59]. They had diameters appropriate for single-mode fiber lasers, but did not have optical quality [48–50,60]. Bend strengths of 0.5 to 1.7 GPa for fiber with grain sizes >1 μm were demonstrated [48,50,60]. There are two obstacles to development of polycrystalline YAG fiber for structural applications: (1) Processing methods for strengths >2 GPa are lacking, despite the reduced grain-size sensitivity of strength in cubic YAG. (2) Structural fiber requires kilometer lengths of fiber tow with ~500 filaments per tow, and scale-up to this production rate is expensive. The two factors are interrelated—incentive to scale-up is absent unless fiber properties are acceptable. Scale-up is not an issue for fiber lasers, but the defect elimination required for optical quality is far more stringent. Defects that degrade optical quality are also flaws that degrade strength. This is evident for optical glass fibers, which have strengths greater than 5 GPa [61–63]. Polycrystalline YAG fiber lasers require processing techniques for optical quality single fibers <30 μm in diameter with lengths on the order of meters.

Despite the anticipated advantages, polycrystalline YAG fiber lasers have not yet been demonstrated, despite some attempts [64,65]. Optical quality polycrystalline YAG fibers with waveguide losses <1 dB/m necessary for reasonable power output have not been made [4,64]. Processing methods for polycrystalline YAG fibers and relationships between processing methods, microstructures, and fiber properties are discussed. Emphasis is on processing methods used to achieve fiber mechanical properties that are acceptable before thorough optical characterization can be considered. Preliminary reports on this work have been published [66–69]. Further processing refinements used to make optical quality fiber are the subject of ongoing research, and will be the subject of a forthcoming paper.

2. Experiments

2.1. Processing

Fibers were prepared with commercially available undoped YAG powder (Nanocerox, Ann Arbor, MI), Methocel™ (E4M grade, Dow, Midland, MI) as a binder, glycerol (Sigma-Aldrich, St. Louis, MO) as a plasticizer, and deionized water. Chemistry of Methocel™ E4M grade is hydroxypropyl methylcellulose ether. The as-received YAG powder was agglomerated and required ball-milling using alumina balls for 24 h in deionized water. Alumina contamination took place during ball-milling. Mass reduction of the alumina balls was ~0.02 wt%. The particle-size of alumina contaminates formed in this manner is not in the nano-size range [70]. Since the densities of YAG powder and alumina were similar but sizes were different, they could be separated by centrifugation at 5000 rpm. The powders were classified to eliminate particles larger than 0.8 μm with a centrifuge (Super T21, Sorvall, UK) after ball milling. Fibers prepared with and without particle classification were compared. Impurity concentrations of as-received powder were analyzed with glow discharge mass spectroscopy (GDMS) and the results of selected elements are shown in Table 1.

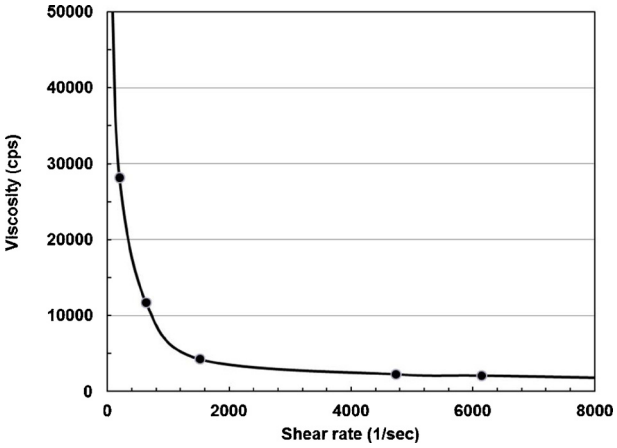

Fig. 1. Typical viscosity vs. shear rate relation of an extrusion mixture.

Methocel™ was obtained as dry powder and dissolved in deionized water as directed in the product manual to form a clear solution. The Methocel™ solution was passed through a 0.45 μm filter to remove anomalously large, undissolved particles. The YAG powder was heat-treated at 700 °C in air before ball-milling to remove organic contaminants. Fibers prepared with and without binder solution filtering and powder heat-treatment were compared. Extrusion mixtures were prepared with ball-milled and classified YAG powder (60–65 wt%), binder (3–5 wt%), plasticizer (2–4 wt%) and deionized water (25–35 wt%). Mixtures of appropriate viscosity were made using a planetary vacuum mixer (ARV-310, Thinky, Japan). They were extruded at 20–35 MPa pressure with a high pressure syringe pump (100 DM, Teledyne, Lincoln, NE) through a 50 μm diameter nozzle. Flow rate (Q) and applied pressure (ΔP) were monitored. This was used to calculate the viscosity (μ) and shear rate (γ) of the extrusion mixtures:

$$\mu = \frac{\pi R^4 \Delta P}{8QL} \quad (1)$$

$$\gamma = \frac{R\Delta P}{4\mu L} \quad (2)$$

where R is the nozzle radius and L the nozzle length. It was assumed that the mixture did not slip on the wall, and flow was fully developed due to small Reynolds number. Fig. 1 shows the viscosity—shear rate relationship of a mixture used for extrusion. All mixtures exhibited shear thinning, which is favorable for fiber formation.

The extruded green fibers were dried at room temperature. In some cases, dried green fibers were cold isostatically pressed at 275 MPa to improve green density before firing. The dried fibers were placed between plastic films and vacuum bagged for cold isostatic pressing (CIP), followed by binder burnout and densification.