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Effect of hot acid etching on the mechanical strength of ground YAG laser elements

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Abstract

High-power pumped Nd:YAG elements may exceed their tensile strength under high thermally induced stress. Providing extra strength to such rods is essential for their employment in high-power lasers. The tensile strength of YAG elements was increased by chemical etching in concentrated phosphoric acid. The highest tensile strength was achieved by etching of fine-ground YAG components: an average $\overline{\sigma}_f = 1130$ MPa for slabs, and $\overline{\sigma}_f = 1225$ MPa for rods, which are 3.6 times and 5 times higher than those of non-etched elements, respectively. The measurements were carried out by four-point flexure strength test. We have established a dependency among the micro-roughness of YAG elements, the surface morphology obtained by etching, and the tensile strength: the tensile strength of the etched element improves for finer after-etch surface texture, which is obtained for finer initial micro-roughness.

To assure the withstanding of Nd:YAG rods under high thermal gradients, a new approach was employed, namely, increasing the pump-power applied to the Nd:YAG rod till fracture. Our results show an increase by more than 2.7 times in tensile strength of etched Nd:YAG rods as compared to standard commercial rods, which corresponds to a thermal loading of excess of 434 W/cm.

Keywords: A. Optical materials; A. Oxides; D. Fracture; D. Mechanical properties; D. Optical properties

1. Introduction

Nd-doped $Y_3Al_5O_{12}$ (Nd:YAG) single crystals are often fabricated and used as laser gain media. Optical pumping of laser rods is usually accompanied by a simultaneous active cooling through its circumference. This induces considerable tensile stresses inside the rod, which may lead to fracture. This determines the upper limit for the average laser output power obtainable. The theoretical tensile strength of a defect-free material is given by the expression [1]

$$\sigma_f^{(th)} = \sqrt{\frac{E\gamma_{SE}}{d_0}} \approx \frac{E}{10}, \tag{1}$$

where E is Young's modulus, γ_{SE} is the surface tension coefficient, and d_0 is the spacing between planes of atoms in the crystal. Taking the Young modulus of YAG as 310 GPa [2a], its theoretical tensile strength may exceed 31 GPa. This value is more than two orders of magnitude larger than the actual YAG tensile strength, quoted as 176–206 MPa [2b].

The figure-of-merit for thermal-stress resistance of a brittle crystal, R_T, is given by [3]

$$R_T = \frac{\sigma_f \kappa (1-\nu)}{\alpha E}, \tag{2}$$

where σ_f is the tensile strength, κ is the thermal conductivity, ν is Poisson's ratio, and α is the thermal expansion coefficient. Notably κ, ν, α and E are all intrinsic material properties, while σ_f depends on its fabrication procedure. The fracture-toughness parameter, K_c, is also an intrinsic property of the material. Standard fracture mechanics relate the tensile strength and the fracture toughness, K_c, by [4]

$$\sigma_f = \frac{YK_c}{\sqrt{a}}, \tag{3}$$

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in which a is the depth of flaws responsible for failure, and Y is a crack orientation and geometry factor, which is of the order of a unity. Eq. (3) indicates that it is possible to improve the tensile strength of brittle materials by reducing the size of the surface flaws [5]. Increasing the tensile strength of Nd:YAG laser rods by chemical etching, which is described in the present work, is based on this very concept [6–9].

Brittle materials such as YAG actually contain flaws of various size and shape. Thus, there is a considerable scattering in measured samples' tensile strength. There is also a large difference between theoretical and actual strength of a brittle material that arises from surface and sub-surface defects, associated mainly with the cutting, drilling, grinding, and polishing processes involved in the element fabrication. In order to design a high-power laser, the probability of rod failure should be accounted. Weibull's theory of brittle fracture [10] provides a statistical approach to assessing a laser rod of durability under high-power lasing.

There is a wide range of strengthening mechanisms that can be used to increase the strength of brittle materials, such as annealing, compressive surface stresses, and fiber reinforcement. The effectiveness of flame polishing and etching for reduction of critical surface flaws has also been stressed [11]. However, the leading technique to improve garnet crystals strength used as laser media is by removing their outer damaged layer. This may be done by laser conditioning, ion beam bombardment, and chemical or plasma etching. Chemical etching is notably simple. It can be performed using standard chemical laboratory equipment, requires relatively short treatment durations, at a low cost. Several chemical etchants are suitable. These include molten KOH at temperatures above 800 °C, concentrated (>40%) hydrochloric acid (HCl) at 250 °C under a relatively high pressure [12], concentrated phosphoric acid [13,14], or alternatively a mixture of phosphoric and sulfuric acids [6]. Chemical etching of YAG in phosphoric acid at moderate temperatures (<220 °C), not only removes the material outer layer, but also reveals dislocation etch-pits [15]. The technique of wet chemical etching was adopted long ago by Koechner to increase the strength of Nd:YAG lasing elements [7]. Etching of Nd:YAG in concentrated H_3PO_4 was also reported by Marion, who increased the strength of Nd:YAG by a factor of 15 [8]. This dramatic result was achieved by wet chemical etching out of a ~75 μm thick layer [8] that was slightly thicker than the damaged layer (50 μm). For strengthening of Nd:YAG components, Shafer et al. used a 1:1 mixture of concentrated sulfuric and phosphoric acids [6].

In the present work, we examine various parameters of mechanical strengthening of crystalline YAG laser components, by wet chemical etching, with the goal of achieving highest durability of the components under high-power pumping.

2. Experimental methods

The experimental setup for acid etching of YAG samples included a resistance-heated furnace, controlled by a Eurotherm controller type 900EPC, equipped with a chromel–alumel thermocouple. The test-tube-like dissolution vessel was made of a thick-walled silica glass, into which another thermocouple protected by a fused silica capillary tube was inserted. It served as a complementary monitor of the solution temperature.

For etching, we used 85% concentrated ortho-phosphoric acid (H_3PO_4) and 95–97% concentrated sulfuric acid (H_2SO_4). The concentrated H_3PO_4 was pre-heated to the desired temperature (180–275 °C) to support evaporation of included water. We used two types of etchants: concentrated H_3PO_4, and a 1:1 mixture (by volume) of H_3PO_4 and H_2SO_4. Etching was initiated by introducing the crystalline sample into the pre-heated acid (or acid mixture), and was terminated by removing the dissolution vessel from the furnace; this resulted in a fast drop in temperature by spontaneous ambient air cooling. After reaching room temperature, YAG elements were rinsed with distilled water. For etching rate determination, the procedure was repeated several times at each temperature, on the same rod. The etching rate R was calculated from the measured weight loss of a sample of known geometry as a function of temperature. The YAG-specific gravity was taken as $\rho = 4.56\,\mathrm{g/cm^3}$.

For etching rate studies we used commercial Nd:YAG laser rods, 4 mm in diameter and ~50 mm long. For the mechanical measurements, undoped YAG slabs ($3 \times 4 \times 50\,\mathrm{mm^3}$) and rods ($\phi\ 4 \times 50\,\mathrm{mm^2}$) were used, with the rod axis parallel to the ⟨1 1 1⟩ crystallographic direction. Prior to etching, the outer faces of the rods and slabs were grounded to achieve a desired micro-roughness. In the present work two degrees of the micro-roughness were used, consistent with the grinding tools availability: >N8 (>3 μm) and ~N6 (~0.8 μm), to be termed later hereafter as "ground" and "fine-ground", respectively. The micro-roughness of the crystalline samples was measured using a Rodenstock profilometer type RM-600-S. The samples were then etched at different temperatures for different lengths of time. For each etching run, a new pre-heated phosphoric acid batch was used.

Four-point flexure strength test was carried out using an Instron Model 1342 load frame, at a displacement rate of 0.5 mm/min. The maximum tensile strength of a slab, σ_{max} was calculated by [7]

$$\sigma_{max}^{slab} = \frac{3FL_o}{4bd^2}, \tag{4}$$

where F is the fracture load, L_o is the outer span, b is the slab width, and d is its thickness. For YAG rods of radius r, the maximum tensile strength was calculated by [7]

$$\sigma_{max}^{rod} = \frac{2FL_o}{\pi r^3}. \tag{5}$$

Results obtained by flexure strength test were analyzed by the Weibull distribution statistics [1]. This allows estimating the failure probability of brittle components under applied stress. In Weibull analysis, the failed samples