

# Physical aspects of laser nitriding of yttria stabilized t-zirconia

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## Abstract

This manuscript describes the surface nitriding of yttria ( $Y_2O_3$ ) stabilized tetragonal zirconia ( $t-ZrO_2$ ) by using the pulsed Nd-YAG laser irradiation in the nitrogen atmosphere. The results show modification of the crystallographic structure between the original tetragonal zirconia and the treated one. Nitrogen atoms enter in zirconia substituting oxygen atoms in the network. X-ray diffraction and Raman spectra show an efficient transformation of the  $t-ZrO_2$  at the surface that exhibit typical yellow-gold color of ZrN with high hardness and wear resistance. The X-ray diffraction results evidenced the new diffraction lines [ $c-ZrN(2\ 0\ 0)$ ] that appear on the nitrided sample with alteration and shift in peaks as well as change in  $d$ -spacing or lattice parameter, whereas the Raman spectra revealed the two characteristic bands related to acoustic part of low frequency in the  $150\text{--}260\text{ cm}^{-1}$  region and optical part at high frequency in the  $450\text{--}750\text{ cm}^{-1}$  region. Nitriding is accompanied by the formation of a few micron thick gradient layer of ZrN structure on the top via dissociative process:  $2ZrO_2 + N_2 = 2ZrN + 2O_2$  and a partial transformation of  $t-ZrO_2$  into cubic zirconia ( $c-ZrO_2$ ) beneath deep in the bulk due to nitrogen concentration gradient. Some new features on the physical characteristics such as hardness and roughness of the cut sections of  $t-ZrO_2$  in the  $N_2$  and  $O_2$  atmosphere are discussed.

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**Keywords:** Laser;  $t-ZrO_2$ ; ZrN; XRD; Raman microscopy

## 1. Introduction

Zirconia ( $ZrO_2$ ) has been of much interest since its discovery in 1892, because of its characteristic properties of corrosion resistance, thermal shock resistance and a toughening agent [1]. Its crystallographic structure and transformation into various phases are of considerable research interest [2]. At high temperature close to its melting point, it has a cubic structure, where Zr-atom is surrounded by eight oxygen atoms forming a primitive cubic sublattice. However at slightly lower temperature between  $2300$  and  $1170\text{ }^\circ\text{C}$ , there is a slight tetragonal distortion from the cubic structure. And at room temperature, i.e. below  $1170\text{ }^\circ\text{C}$ , its stable phase is monoclinic. This is a further distortion of cubic structure. This transformation may be suppressed by addition of 2–3% mole of yttria ( $Y_2O_3$ ) and shows better mechanical properties. Surface treatment of such materials, i.e. yttria stabilized tetragonal zirconia ( $t-ZrO_2$ ) by

means of high energetic beam in the reactive/dissociative atmosphere of nitrogen is an attractive technique to enhance the surface features such as corrosion, wear resistance and hardness. Nitrogen being very stable and inert under normal conditions, when react with such elements forcing a tangible compound such as nitrides (ZrN). They possess interesting properties, e.g. high melting point, high wear and corrosion resistance and are much beneficial to tool industries especially in coating applications. Besides its use in decorative marking, it can also find application in superconducting devices such as Josephson's junction ( $ZrN/Zr_3N_4/ZrN$ ), etc., and as a diffusion barriers in IC technology due to lowest electrical resistivity and better thermal stability. Several techniques are already in use to modify the surface of zirconia so as to develop a nitride coat on it or any other substrate material [3–8]. Besides this, various kind of lasers have also been used to cut and or effectively modify the surface characteristics of various materials for nitriding in the reactive atmosphere [9–11]. However, using the pulsed Nd-YAG laser has emerged as an attractive technique [12] in nitriding the tetragonal zirconia ( $t-ZrO_2$ ) to a stable nitride (ZrN) phase via dissociative process:  $2ZrO_2 + N_2 = 2ZrN + 2O_2$ , demonstrating the efficient nitrogen incorporation and formation of thick nitrided layer with superior tribological properties. The present work deals with such a study

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<sup>1</sup> Part of this work was done when the author was with Ritsumeikan University and Laser X Co. Ltd., Japan.

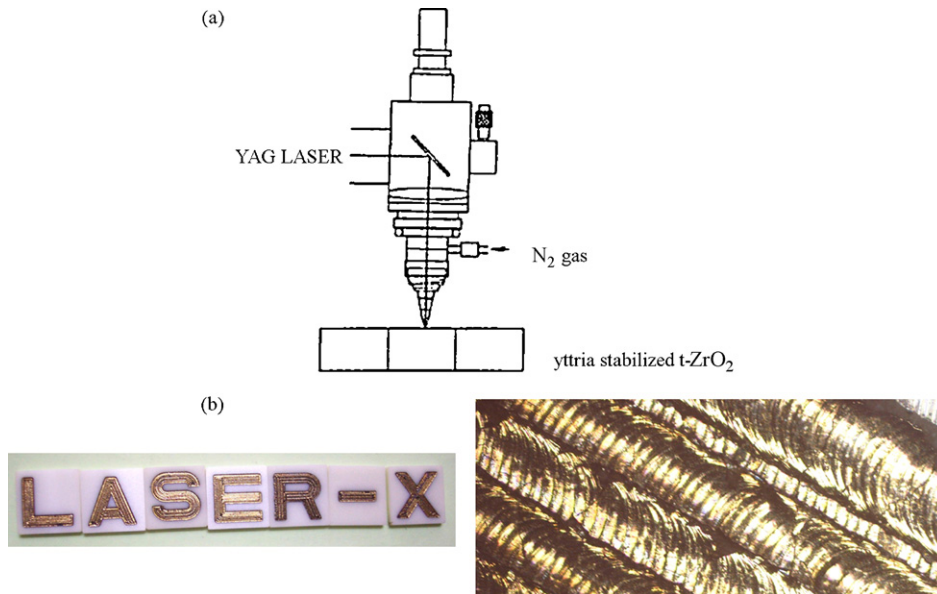


Fig. 1. (a) Block diagram of Nd-YAG laser nitriding of yttria stabilized tetragonal zirconia (t-ZrO<sub>2</sub>); (b) laser scan logo: LASER-X (ZrN: yellow-gold) and a partial magnified view; white un-scanned area is yttria stabilized tetragonal zirconia (t-ZrO<sub>2</sub>). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

and explores the possibility of analyzing the structural properties of the nitrided sample with X-ray diffraction and Raman microscopy. It opens new challenges to obtain structural information of the nitrided (ZrN) and bare tetragonal zirconia (t-ZrO<sub>2</sub>) sample. Examples of the decorative marking applications as well as in improving the cut quality are well illustrated.

## 2. Experimental

The laser nitriding/cutting experiments were carried out using a pulsed Nd-YAG laser (Lumonics: Model: JK701) with experimental details (Fig. 1a) described elsewhere [13]. The beam was focused onto the specimen plate comprised of

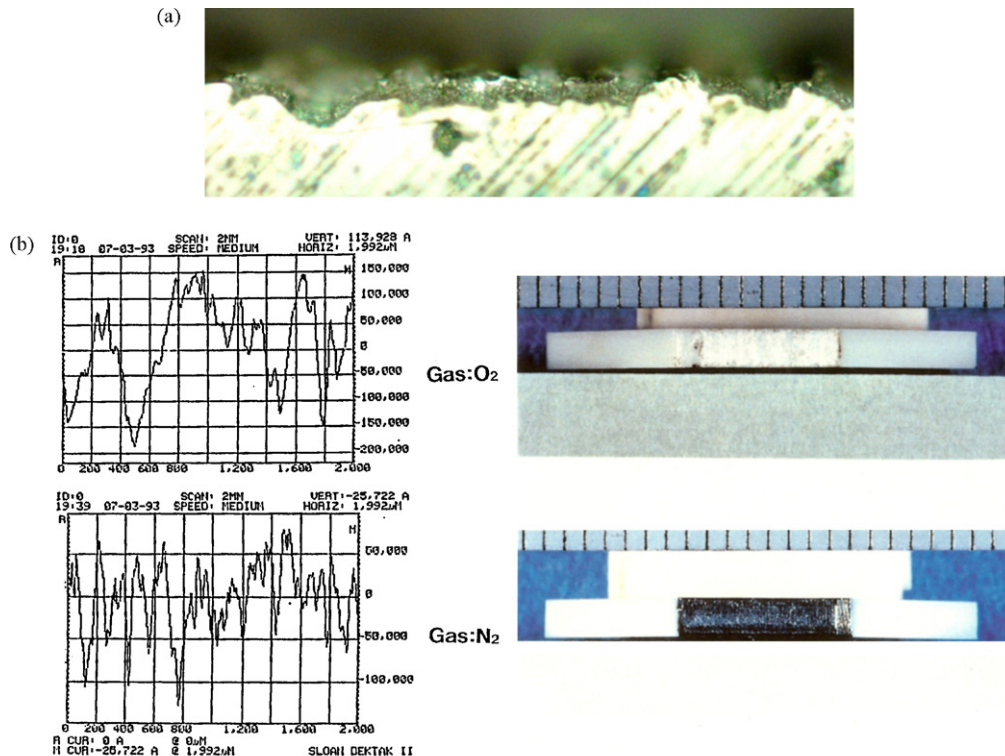


Fig. 2. (a) Laser cut section of t-ZrO<sub>2</sub> transformed into ZrN (thickness of layer = 10 μm); (b) cross-section and roughness profile of the cut surface of t-ZrO<sub>2</sub> in O<sub>2</sub> and N<sub>2</sub> atmosphere.

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