

Ductile Mode Machining of the Micro Pattern Made on YSZ Using Ultra-precision Shaping with a Diamond Tool

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Yttria stabilized zirconia (YSZ), which is a ceramic material, has a number of applications such as a refractory, thermal barrier coating and as a solid electrolyte for a solid oxide fuel cell (SOFC). Micro patterning the YSZ can increase the efficiency of the SOFC, but YSZ is difficult to mechanically machine. A few researchers have reported that an ultra-fine pattern can be mechanically machined on ceramic materials with no brittle fracture with a depth of cut close to sub-micrometers (called ductile mode machining). In the present study, the conditions for ductile mode machining of YSZ were studied. A 3-axis ultra-precision machine system and 90° diamond tool were employed to machine a micro pattern on YSZ. At first, when YSZ was machined with a depth of cut of 1 μm and 10 passes, the micro pattern was entirely fractured due to the brittleness of YSZ. Next, the micro pattern was machined with a depth of cut of 1 μm and 1 pass to verify how multi-pass machining affected the brittle fracture. A sparse brittle fracture occurred, which meant the depth of cut of 1 μm was too large for ductile mode machining. A mix of ductile mode machining and brittle mode machining was observed. Thirdly, when YSZ was machined with a depth of cut of 0.5 μm by 20 passes, the micro pattern was clearly machined with ductile mode machining. Thus, a transition point between ductile mode machining and brittle mode machining should exist at a depth of cut between 0.5 μm and 1 μm . A nanoscratch test was used to determine the transition point. The transition point was found to be 875 nm by analyzing the lateral force and the machined surface.

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I. INTRODUCTION

Yttria stabilized zirconia (YSZ) is a face-centered cubic ceramic whose crystal structure of zirconium dioxide is made stable at room temperature by the addition of yttrium oxide. Because of its chemical inertness and

hardness, YSZ has a number of applications including a refractory, thermal barrier coating in gas turbines and a solid electrolyte for a solid oxide fuel cell (SOFC) [1]. Thin film YSZ is manufactured by using the laser deposition method, and bulk YSZ is manufactured by using the melting method [2,3]. Many studies about the manufacturing methods for YSZ have been done and the efficiency of a SOFC is known to be proportional to the surface area of YSZ. Thus, if surface area of YSZ is in-

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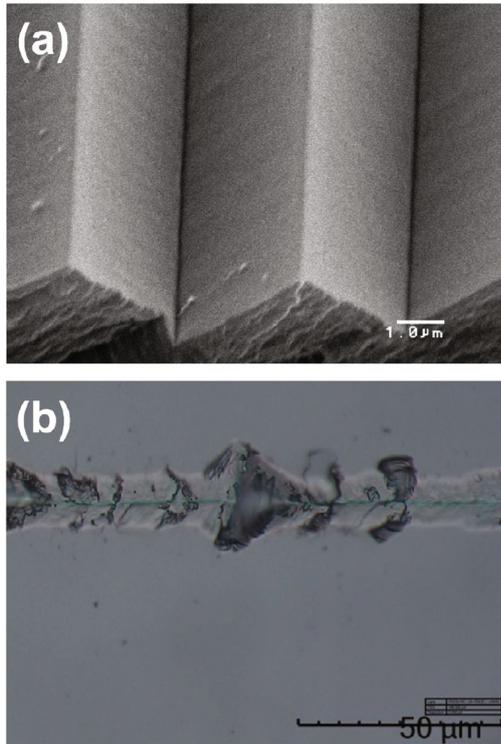


Fig. 1. (Color online) Example of a machined micro pattern (a) on a ductile material and (b) on a brittle material.

created by machining, the efficiency of the SOFC can be increased [4]. If this is to be accomplished, a micro pattern can be machined on the surface of YSZ by using a laser method. However, precise machining of micro patterns by using laser is difficult, and the resulting machined surface is not good [5]. A mechanical machining method is advantageous for precisely machining a micro pattern on metal molds. Due to its brittleness, YSZ, like other ceramic materials, is usually machined by grinding among the various types of mechanical method. Ceramic materials can also be ground with an ultrasonic assisted tool; however, grinding is not suitable for machining a micro pattern because the tool is too large [6]. When ceramic materials are machined by using a diamond tool, a micro groove can be machined with a specific cutting depth [7]. Ductile mode machining can be performed on brittle materials by decreasing the depth of cut. However, repeated machining of micro patterns on ceramic materials by using an ultra-precision machine tool has not been studied much yet. In the present study, a micro pattern was machined on YSZ by using a diamond tool and an ultra-precision system.

II. MICRO PRISM PATTERN MACHINING

Machining a micro pattern on a metal mold by using a diamond tool has already been studied [8]. The shape

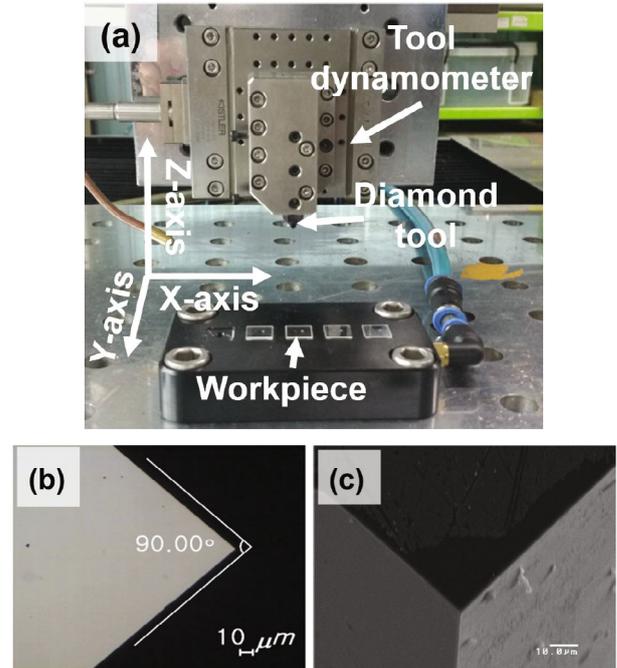


Fig. 2. (Color online) (a) Micro pattern machining system, (b) 90° diamond tool and (c) SEM image of the diamond tool's tip.

of the tool and the depth of cut were determined and applied according to shape of the pattern. The micro pattern was not machined in one-pass, but was machined in multiple passes, which were divided by the proper depth of cut. A micro pattern was machined on a Ni-plated mold by using a 90° diamond tool as shown in Fig. 1(a). The Ni-plated mold could be precisely machined with a depth of cut of 0.5 to 2.5 μm . This machining characteristic appears not only with a Ni-plated mold but also with non-ferrous metal molds because a non-ferrous metal has a ductile property. Thus, the micro pattern on these metal molds can be machined without fracture. However, this is in contrast to the micro patterning of a material that has a brittle property. A micro groove machined on a Si-wafer, which is a brittle material, is shown in Fig. 1(b). The micro pattern fractured at a depth of cut of only 2 μm because the Si-wafer was a brittle material. In the present study, because YSZ is also brittle, micro patterning of YSZ was machined after considering the possibility of fracture.

III. EXPERIMENTS AND DISCUSSION

A single-crystal YSZ workpiece composed of Y_2O_3 stabilized ZrO_2 (8% mole Y_2O_3) was fabricated with a size of $10 \times 10 \times 1 \text{ mm}^3$. Both sides of the sample were polished by using chemical mechanical polishing (CMP), which produced a surface roughness under 5 \AA . An ultra-precise 3-axis machine system was employed for the ex-

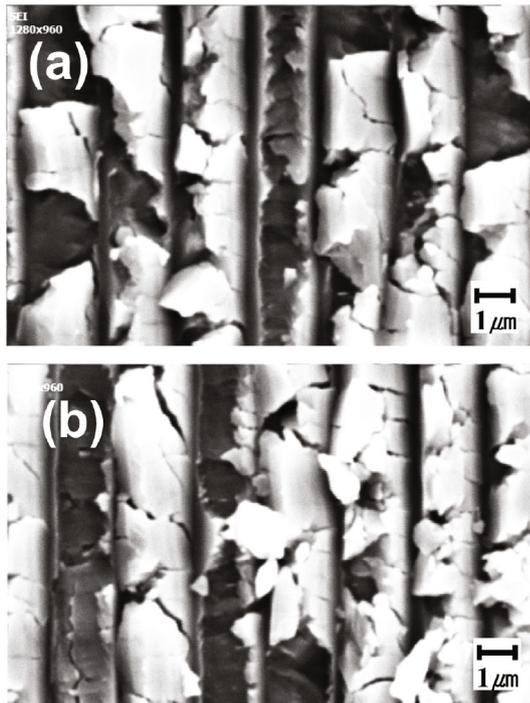


Fig. 3. Machined micro pattern with a depth of cut of $1\ \mu\text{m}$ made by using multi-pass machining at (a) the entry area and (b) the middle area.

periment. Each axis had a $5\ \text{nm}$ resolution and was driven by linear motors, as shown in Fig. 2(a). A tool dynamometer (Kistler 9256C2) was installed on the X-Z axis to measure the cutting force precisely and to touch the workpiece. A single-crystal diamond tool was employed, the tool shape angle was 90° , and the radius of the tool tip was under $100\ \text{nm}$, as shown in Fig. 2(b).

The machined surface was measured and analyzed by using scanning electron microscopy (SEM) and atomic force microscopy (AFM). The depth of cut was $1\ \mu\text{m} \times 10$ passes, the cutting pitch was $2\ \mu\text{m}$, and the cutting speed was $200\ \text{mm/s}$. The most important cutting condition with respect to the potential for brittle fracture is the depth of cut. The cutting area is determined by the depth of cut, so a ductile mode or a brittle mode is determined. In the first experiment, the depth of cut was set at $1\ \mu\text{m}$ based on an existing paper, which reported ductile mode machining of a ceramic material [9]. The cutting was divided into 10 passes because the micro cutting was conducted by over-depth due to the flatness of the workpiece.

Following the cutting, an entry area and a middle area of the machined YSZ were measured by using SEM, as shown in Fig. 3. The entire area of the machined pattern on the YSZ was clearly fractured. This was a result of brittle mode machining due to the large depth of cut, also likely multi-pass machining. Multi-pass machining can cause surface hardening or residual stress. As a result, the property of the machined YSZ can be changed

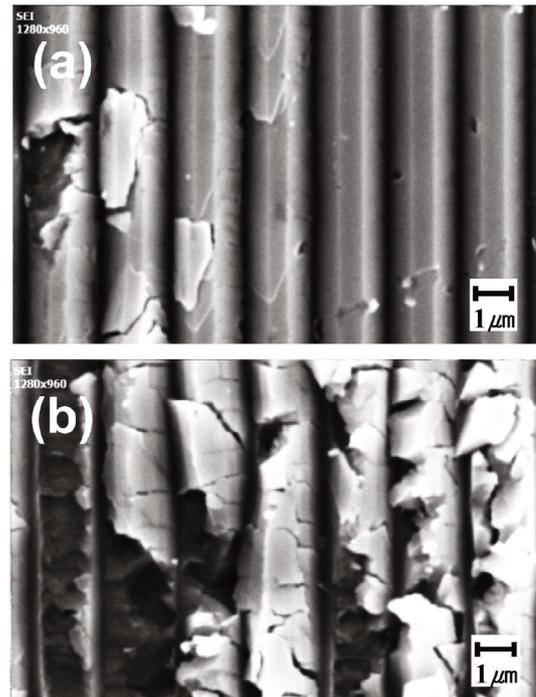


Fig. 4. Machined micro pattern with a depth of cut of $1\ \mu\text{m}$ made by using single-pass machining at (a) the entry area and (b) the middle area.

in comparison with the initial YSZ. To verify this, we conducted single-pass machining with a depth of cut of $1\ \mu\text{m}$. The other cutting conditions were kept the same as for the prior experiment. Likewise, the entry area and the middle area of the machined YSZ were measured by using SEM, as shown in Fig. 4. Unlike the first experiment, the fractured pattern and a clearly-machined pattern simultaneously appeared. Beyond the middle area, the micro pattern was fractured, just as in the first experiment. Thus, the brittle mode machining was mostly effected by the depth of cut, and the multi-pass machining made brittle mode machining in the entire machined area.

In the third experiment, the depth of cut was $0.5\ \mu\text{m}$, and the YSZ was machined in 20 passes. After 20 machining passes, the total depth of cut was the same as it was in the first experiment. The other cutting conditions, such as the cutting speed and the cutting tool, were same as they were in the first experiment. After micro pattern machining, the machined micro pattern was measured by using SEM as shown in Fig. 5. The micro pattern was clearly machined in the entry and the middle area. Ductile mode machining appeared at a depth of cut of $0.5\ \mu\text{m}$, and multi-pass machining was shown to have no influence on the ductile mode machining. The machined YSZ was measured by using AFM in order to precisely analyze the micro pattern. The measured pitch of the prism pattern was almost $1\ \mu\text{m}$, and nano fractures were sparsely observed.

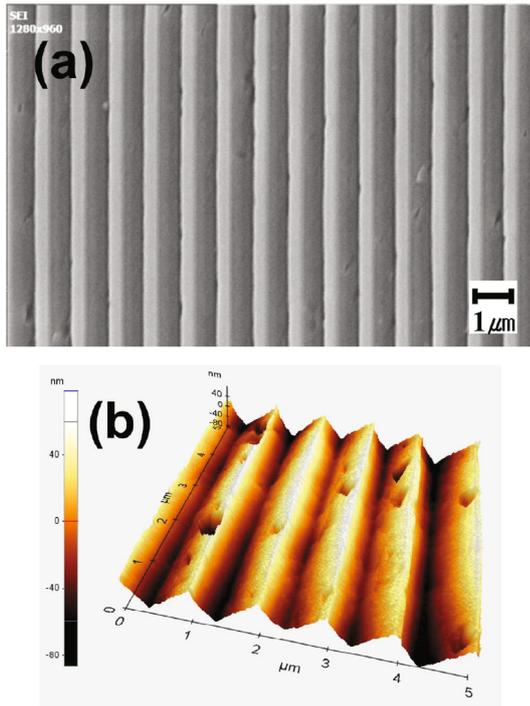


Fig. 5. (Color online) Machined micro pattern with a depth of cut of $0.5 \mu\text{m}$ made by using multi-pass machining (a) a SEM image and (b) an AFM image.

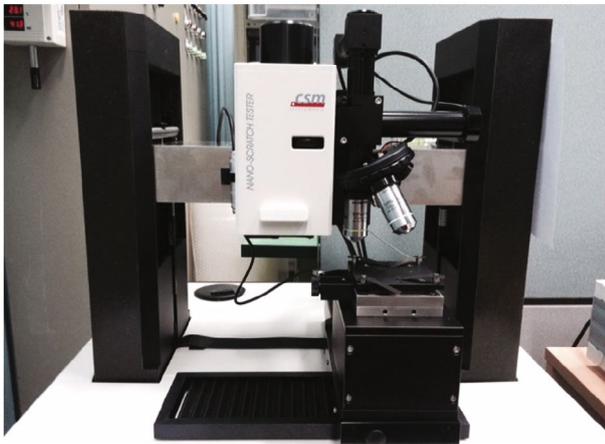


Fig. 6. (Color online) Nanoscratch tester used for measuring the in-situ cutting force and the depth of cut.

YSZ, like a metal, can be machined in the ductile mode with a depth of cut of $0.5 \mu\text{m}$. However, measuring the limit point of ductile mode machining by using just an observation of the machined pattern was difficult. Thus, the limit point of the ductile mode machining was measured by using a nanoscratch tester which was not applied to machine the pattern but was able to measure in-situ the cutting force and the depth of cut [10]. The nanoscratch tester (Anton-Paar, NST-Z-CE) employs a standard cantilever, a standard friction table, and optical inspection, as shown in Fig. 6. Specifications were a

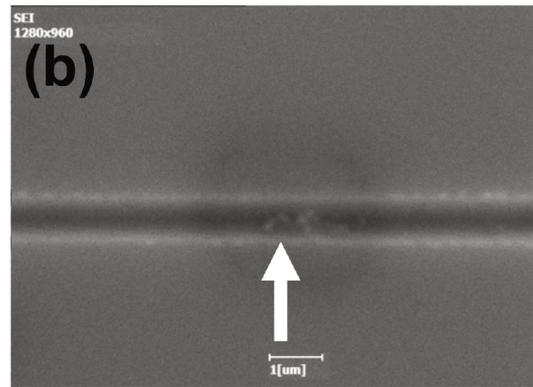
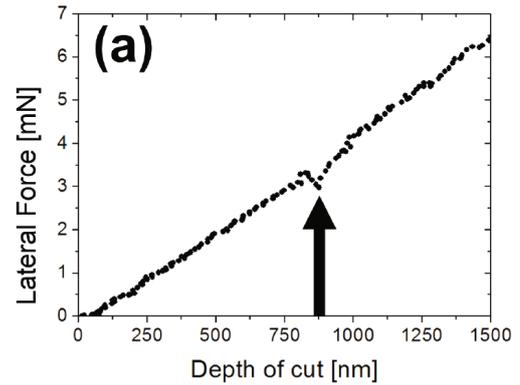


Fig. 7. (a) Variation of the lateral force versus the depth of cut and (b) SEM image of the machined pattern at the first fluctuation point.

normal force from 0.1 to 100 mN, a lateral force of 0 – 1 N, loading rates of 0 – 10 N/min, a scratch length of 0 – 60 mm, and a speed (X-axis) of 0.4 – 600 mm/min. The normal force corresponding to a depth of cut of $1 \mu\text{m}$ from the prior test was applied because this test was controlled by using the normal force. A normal force from 0 to 110 mN was applied, and the cutting speed was 1 mm/min. The normal force, the lateral force, and the cutting depth were measured while machining the micro pattern. The variation of the lateral force versus the depth of cut and a SEM image of the machined pattern at the first fluctuation point are shown in Fig. 7. The first fluctuation point of the lateral force was at a depth of cut of 875 nm. The machined pattern was matched with the lateral force data. The machined pattern was fractured at the fluctuation point. Thus, ductile mode machining of YSZ changed to brittle mode machining at a depth of cut of 875 nm.

IV. CONCLUSION

In the present paper, a micro pattern was successfully machined on YSZ by using a diamond tool. The first machined micro pattern on YSZ with a depth of cut of $1 \mu\text{m}$ was fractured as in brittle mode machining. On the

other hand, the micro pattern on YSZ with a depth of cut of $0.5\ \mu\text{m}$ was clearly machined as in ductile mode machining. A nano fracture was sparsely observed in spite of ductile mode machining. Ductile mode machining of YSZ changed to brittle mode machining at a depth of cut of 875 nm.

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REFERENCES

- [1] N. O. Nwosu, A. M. Davidson and C. S. Hindle, *Adv. Chem. Eng. Sci.* **1**, 118 (2011).
- [2] S. H. Kim and B. H. Park, *J. Korean Phys. Soc.* **47**, S247 (2005).
- [3] T. Sakuma, Y. Yoshizawa and H. Suto, *J. Mat. Sci.* **20**, 2399 (1985).
- [4] P. C. Su and F. B. Prinz, *Electrochem. Commun.* **16**, 77 (2012).
- [5] J. Parry, R. Ahmed, F. Dear, J. Shephard, M. Schmidt and L. Li, *Int. J. Appl. Ceram. Tech.* **8**, 1277 (2011).
- [6] H. Huang, *Mat. Sci. Eng. A* **345**, 155 (2003).
- [7] Y. Kamimura, H. Yamaguchi and Y. Tani, *Ann. CIRP* **46**, 451 (1997).
- [8] T. J. Je, S. C. Park, K. W. Lee, Y. E. Yoo, D. S. Choi, K. H. Whang and M. C. Kang, *Trans. Nonf. Metals Soc. China.* **19**, s288 (2009).
- [9] J. C. Morris, D. L. Callahan, J. Kulik, J. A. Patten and R. O. Scattergood, *J. Am. Ceram. Soc.* **78**, 2015 (1995).
- [10] J. Valli, *J. Vac. Sci. Tech. A* **4**, 3007 (1986).