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Thermal effect on brittle–ductile transition in CaF_2 single crystals

Akshay Chaudhari, Yan Jin Lee, Hao Wang*, A. Senthil Kumar

Department of Mechanical Engineering, National University of Singapore, EA-07-08, 9 Engineering Drive 1, Singapore 117575

mpewhao@nus.edu.sg

Abstract
Single crystal calcium fluoride (CaF_2) is widely used to manufacture optical lenses due to its excellent optical properties. Conventionally CaF_2 is fabricated using grinding and lapping for aspherical and free form optics. However, these fabrication processes are time-consuming and difficult to control; hence single crystal diamond turning is often used to fabricate optical components with the nanoscale surface finish. The diamond turning process is conducted at the optimal uncut chip thickness to ensure ductile mode machining of the brittle CaF_2 single crystals. In general, the ductile mode machining of CaF_2 is performed at an extremely low uncut chip thickness (<100 nm) which adversely increases the machining time and cost. This work proposes a novel method to potentially increase the critical uncut chip thickness by modifying the brittle–ductile transition characteristics through elevating the temperature of CaF_2 crystals. Three crystallographic planes, (111), (110), and (100), have been studied in microhardness tests under different heating conditions. A significant decrease in hardness has been observed for all the crystallographic orientations as the temperature increases. Further observations on the indentation morphology indicate that crack formation can be suppressed at elevated temperatures, which implies a thermally enhanced plasticity of CaF_2 single crystals.

Keywords: calcium fluoride, brittle–ductile transition, thermal effect, indentation, microhardness

1. Introduction

Calcium fluoride (CaF_2) single crystals are widely used in optical instruments due to its wide transmission range (180 nm to 8 μm) and high Abbe number (95.1). However, being a brittle crystal with anisotropic properties possesses multiple challenges in the fabrication of CaF_2 components. The fabrication process such as grinding is time-consuming whereas in laser machining, fracture and cracks in the single crystal structure is caused by thermal shock [1]. Moreover, complex shapes cannot be fabricated by etching due to the inherent material anisotropy of CaF_2 single crystals [2]. Hence, ultra-precision diamond turning is used to fabricate complex freeform optical lenses. The diamond turning process of CaF_2 is based on the ductile–brittle transition of the material at a sub-micron uncut chip thickness. Although the ductile mode machining of CaF_2 single crystals is possible, it is mainly incapacitated by the subsurface damages and microstructural changes on the machined surface. Moreover, the uncut chip thickness changes significantly, with the crystallographic orientations and cutting directions, ranging from 30 nm to 380 nm as reported by Wang et al. [3]. The variation in uncut chip thickness not only makes it difficult to optimise the cutting parameters but also leads to unstable cutting conditions due to the variation in thrust forces.

In this study, we explore the possibility of increasing the uncut chip thickness and suppressing crack formation with thermally enhanced plasticity in CaF_2 crystals. The improved plasticity was evaluated by micro-indentation tests at elevated temperatures.

2. Experiments

Experimental setup as shown in Figure 1 was used to elevate the temperature of CaF_2 single crystal samples of the size 10 mm x 10 mm x 5 mm. The increase in temperature was recorded using an infrared (IR) camera. The emissivity coefficients for the IR camera were calibrated using thermocouple temperature measurements of CaF_2 crystals. Three different crystallographic planes, (100), (110), and (111), were used in this investigation to gain better understanding of the material behaviour and crack initiation mechanism. Vickers microhardness tests were performed at four temperatures as 23.5 °C (room temperature), 50 °C, 75 °C, and 100 °C, respectively. Moreover, variation in temperature was kept within ±5 °C. For this investigation, a load force of 0.5 N load was applied to the sample surface with the indenter travelling at 0.5 mm/s and a dwell time of 15 s. Three indentations tests were conducted at each of the designated temperatures and the average hardness values are reported in this paper. The crack formation around the indentation markings was evaluated using an optical microscope and a scanning electron microscope (SEM). An ultra-thin layer (5–22 nm) of gold was applied on the CaF_2 samples using the sputter coating method to ensure a higher resolution of the captured SEM images which were used for qualitative evaluation.

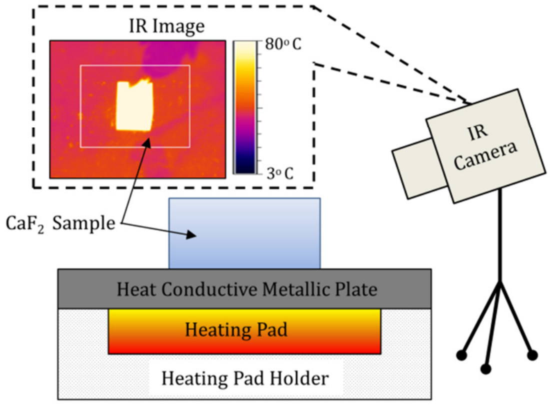

Figure 1. Experimental setup for temperature elevation and measurement of CaF_2 samples.

3. Results and discussions

The microhardness test results of CaF_2 single crystals at four designated temperatures ranging from 23.5 °C to 100 °C are presented in Figure 2. A significant decrease in hardness with increase in temperature can be observed irrespective of the crystal orientations. For the given temperature range, there is a loss of ~50% in hardness.

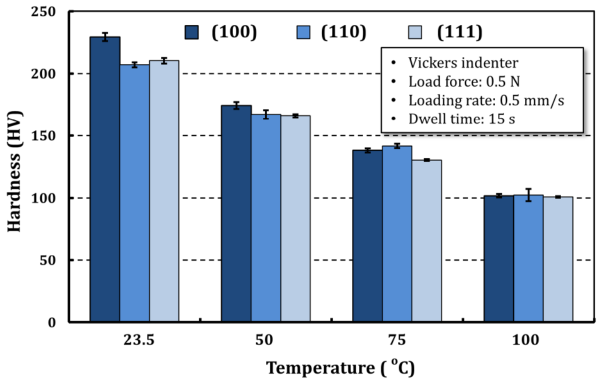

Figure 2. Decrease in hardness of CaF_2 single crystals with increase in temperature.

For indentation on the (110) plane, there are cracks emanating from the indenter whereas such cracks are absent for indentation on the (100) and (111) planes even at lower temperatures as indicated in Figure 3. The (110) CaF_2 crystal is prone to crack formation as the cleavage plane coincides with slip planes as shown in Figure 4(b). The load force as small as 0.5 N is sufficient to initiate crack formation on the (110) plane whereas higher load forces are required to induce crack formation on the other two crystallographic planes. These observations bear close resemblance to the one reported by Azami et al. [4]. They found that the crack emanated at load forces of 0.5, 1, and 5 N on the crystals with (110), (111), and (100) orientations, respectively. The effect of variation in dislocation behaviour with regard to different slip systems is also reflected by the hardness values, for example, high hardness was observed for the (100) plane as the angle between the loading direction and cleavage plane is higher (Figure 4(a)). The angle further reduces as depicted in Figure 4(c) for the (111) plane leading to a decrease in hardness.

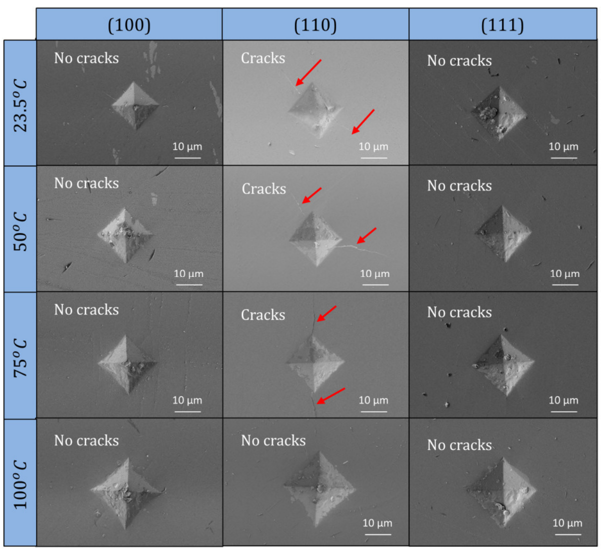

Figure 3. SEM images of indentation markings on the (100), (110), and (111) crystallographic planes at the room and elevated temperatures.

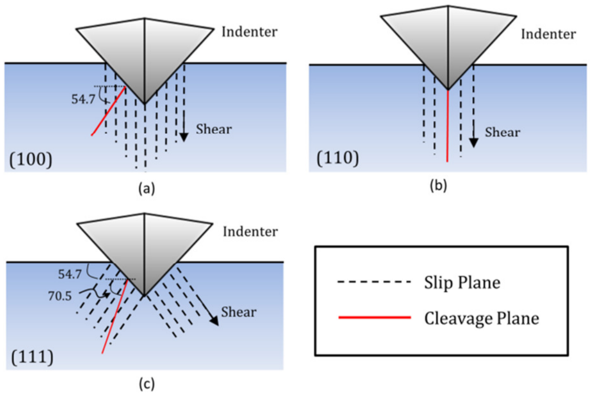

Figure 4. Slip planes and cleavage planes for CaF_2 single crystals: (a) the (100) plane, (b) the (110) plane, and (c) the (111) plane.

It is interesting to note that the hardness values converge to a single value at 100 °C, which indicates that suppression of the crack formation at the elevated temperatures can be achieved irrespective of the crystal orientations. The observed decrease in hardness can be attributed to the activation of more than one slip system as observed by Muňoz et al. [5]. These results are also consistent with our previous work where crystal plasticity finite element method (CPFEM) was used to simulate the micro-cutting of CaF_2 crystals at the elevated temperatures [6]. The simulation results indicated that at elevated temperatures not only the lateral force became symmetric but also the cutting and thrust force dropped by 50% which could be attributed to increased plasticity and reduction in hardness.

4. Summary

In this study, the variation in hardness of CaF_2 single crystals was evaluated on the three different crystallographic planes at the room and elevated temperatures. It was found that the hardness decreases rapidly in addition to a significant suppression of crack formation at the elevated temperatures. Activation of more than one slip system at elevated temperatures leads to the enhanced plasticity. These new findings combined with the simulation results obtained using CPFEM have further validated our proposed method to improve the ductility of the brittle CaF_2 single crystals, i.e. to facilitate the brittle–ductile transition.

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