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Fabrication of an atomically smooth polycrystalline surface without grain boundary steps using catalyst-referred etching

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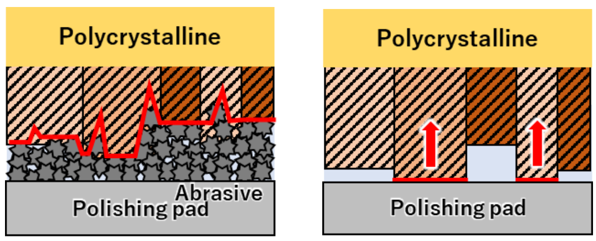
Abstract

Recently, polycrystalline material can express its excellent physical properties and has been used in advanced applications in semiconductors and optics. Similar to single crystal materials, to fully exploit the properties of polycrystalline material in devices, a flat surface on the atomic level is required. However, since the polycrystalline material surface has various crystal orientations, the grain boundary steps appear on the surface polished by CMP (chemical mechanical polishing). Thus, new polishing technology needs to be developed. The following two conditions are required to obtain atomically well-ordered polycrystalline material surfaces: 1) The polishing pad must always act as a reference flat to selectively remove the crystal grain located at the topmost of the sample surface. 2) The material removal must proceed via a chemical etching to avoid crystallographic damages. Therefore, we developed a chemical etching method assisted by a metal catalyst named catalyst-referred etching (CARE). In this method, a polishing pad with a catalyst thin film works as a reference plane to be copied onto the workpiece surface. A workpiece is pressed to the polishing pad in an etchant, and they rotate around their axes. Accordingly, only the grain located at the topmost site frequently contacts the catalyst and is preferentially removed chemically, leading to obtaining a damage-free surface without grain boundary steps. In this study, we demonstrated the possibility of realizing the atomically flat polycrystalline surface by applying the CARE. Polycrystalline SiC and YAG ceramics were processed using Ru catalyst and a solution having neutral pH as a catalyst and an etchant, respectively. Using this method, we obtained an extremely smooth surface with a grain boundary step less than 1 nm without introducing crystallographic damage.

Polycrystalline materials, polishing, chemical wet etching, grain boundary steps free

1. Introduction

Polycrystalline materials have been used as a substitute for single crystal materials because they are inexpensive to manufacture and can be molded into any shape and size. Improvements in growth technology have made it possible to express its excellent physical properties and has been used even in advanced applications in the fields of semiconductors and optics. Similar to functional single crystal materials, to fully exploit the properties of polycrystalline materials in devices, a flat surface at the atomic level is required[1,2]. Chemical mechanical polishing (CMP) is used for polycrystalline widely as the same as for single crystalline. In CMP, a chemical modifies the surface and an abrasive removes the modified surface mechanically. Since the surface has various crystal orientations, the material removal efficiency is different for each crystal grain. The polished surface has many scratches and grain boundary steps, as shown in Fig. 1(a). In recent years, in order to suppress the chemical etching effect and improve the transferability of the reference plane, the use of a nanodiamond and metal plate as an abrasive and polishing pad, respectively, has been proposed[3,4]. However, even if the chemical etching effect is eliminated, it is difficult to maintain a high transferability because the surface of the metal plate is roughened upon contact with a high-hardness abrasive. As a result, the appearance of grain boundary steps and mechanical damage are fundamentally unavoidable. The fabrication of a polycrystalline material surface without grain boundary steps and mechanical damage has not been realized, and a new polishing technology needs to be developed. Based on the above discussion, the following two conditions must be satisfied to obtain atomically well-ordered polycrystalline material surfaces: 1) The polishing pad must always act as a reference flat to selectively remove the crystal grain located at the topmost of the sample. 2) The material removal must proceed via a chemical etching to avoid crystallographic damages. Therefore, we developed a novel polishing method named catalyst-referred etching (CARE), which is based on chemical wet etching assisted by a metal catalyst[5]. The CARE instrument is nearly the same as that used for chemical mechanical polishing. In this method, a polishing pad having a catalyst thin film on the surface is employed. A workpiece is in contact with the polishing pad while rotating in an etchant. The topmost site frequently in contact with the pad is preferentially removed. The polishing surface as a reference plane to be copied to the sample surface, leading to efficient



(a) Chemical mechanical polishing

(b) ideal polishing

Figure 1 Schematic of polycrystalline material polishing

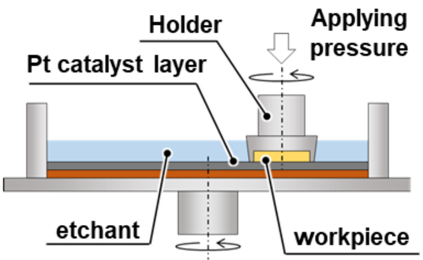


Figure 2 Schematic of CARE instrument

planarization with averaging effect. We have successfully fabricated an atomically flat surface on single crystalline material such as SiC, GaN[6,7]. These surfaces have a step-terrace structure with one atomic layer height due to step flow etching. The etching mechanism has clarified a hydrolysis reaction assisted by the catalyst surface, using first-principles calculations[6,8]. The catalyst assists the dissociation of H_2O and generates OH^- with a high nucleophilicity. The OH^- attacks the step-edge atoms, leading to cleavage of the backbond.

We used the polycrystalline SiC substrate as the sample in this research. It is used as a handling substrate for crystalline SiC, leading to cost down of a SiC substrate. Whereas the conventional polishing method increases the surface roughness which is too large to form a strong bonding[8]. For industrial use, the surface roughness should be decreased to 1.0 nm rms (root mean square). In this study, we demonstrate the possibility of realizing the atomically flat polycrystalline SiC surface by applying CARE.

2. Experiment

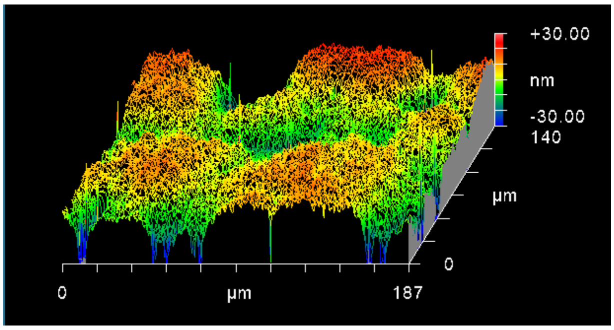
The experimental setup is shown in Fig. 1. The workpiece was placed inside the holder and pushed onto the polishing pad with controlled pressure. Pressure and relative speed between the surfaces of the workpiece and catalyst were 15 kPa and 100 mm/s, respectively. Pt and deionized pure water were used as a catalyst and an etchant. The workpiece was polycrystalline SiC with a polished surface by CMP. Scratch marks produced by a diamond pen were used as reference points on the sample surface, and the same location of the sample surface was measured before and after CARE processing using phase-shift interference microscopy (ZYGO NewView 200 HCR). The appearance of the grain boundary steps was evaluated by observation through atomic force microscopy (AFM, SHIMADZU 9700HT).

3. Result and discussion

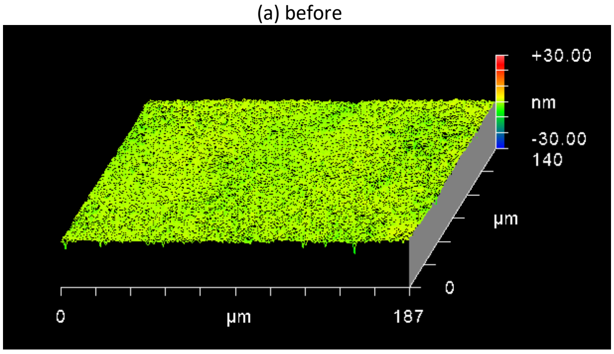
The optical profiles of polycrystalline SiC before and after CARE are shown in Fig 2(a) and (b), respectively. The introduced scratch and boundary steps in CMP processing were completely removed, resulting in decreasing the surface roughness from 14.665 nm to 0.942 nm. Figures 3 are AFM images before and after CARE. We succeeded in obtaining an extremely smooth surface with a grain boundary step of 1 nm or less without introducing crystallographical damage.

4. Conclusion

We proposed applying a novel polishing method which is named CARE to polycrystalline material. In CARE, most of the top grain on the surface contacts with the catalyst layer frequently and is removed chemically, leading to a flat surface. Applying CARE to polycrystalline SiC, we succeeded in fabricating the atomically smooth surface without crystallographical damaged

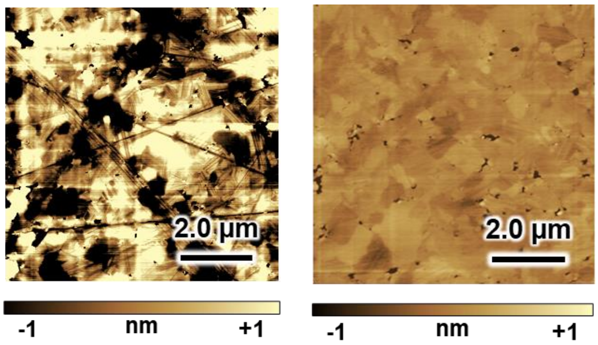


(a) before



(b) after CARE

Figure 2 Optical profile images of (a) pre-processed surface (PV: 964.597 nm, rms: 14.665 nm), and (b) CARE-processed surface (PV: 23.240 nm, rms: 0.942 nm) of polycrystalline SiC



(a) before (b) after CARE

Figure 3 AFM images of (a) pre-processed surface (PV: 4.888 nm, rms: 0.489 nm), and (b) CARE-processed surface (PV: 1.150 nm, rms: 0.132 nm) of polycrystalline SiC

and the grain boundary steps. It can be said that the effectiveness of the CARE method as a surface treatment method for polycrystalline materials has been fully demonstrated. In the future, we plan to fabricate a device using a polycrystalline material substrate polished by CARE, evaluate its characteristics, and investigate the relationship between device characteristics and substrate surface accuracy.

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