

Mechanical Behavior of PCS-derived Si-C Ceramics Strengthened using PIP Method



SiC ceramics model before and after PIPs

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Abstract

Strength evaluation of silicon carbide (Si-C) ceramics fabricated from polycarbosilane (PCS) precursor is described. Si-C ceramics was prepared by firing a green body made of the mixture of Si-C nano-powders and a PCS solution at 1273 K in N_2 gas for an hour. To obtain dense Si-C, the polymer infiltration and pyrolysis (PIP) process was conducted up to 12 cycles. Si-C ceramics was diced to be rectangle shape, and was subjected to the three-point bending test. Si-C specimens fabricated through PIP processes less than 2 cycles showed non-linear force-displacement curves, whereas those through the processes more than 3 cycles showed linear relations and fractured in a brittle manner. The Young's modulus of 12-cycles-PIPs specimen was 56 GPa, 22-fold of non-PIP specimen. The bending strength was also increased up to 157 MPa with an increase in the number of PIP process. The cause of the influence of PIP process on the mechanical characteristics is discussed using a PCS-derived Si-C model.

Experimental Procedure



drastically increased to 68 GPa and 157 MPa. Before PIP, successive brittle fractures of pyrolyzed linkages probably appeared as "apparent" ductility in the force-displacement relations. After PIPs, impregnated PCS was fired many times; consequently, many of unnecessary atoms included in the PCS vaporized, and the pyrolyzed PCS layer definitely toughened. Therefore, a large number of PIP process yielded PCS-derived Si-C ceramics having better mechanical characteristics.

PIP Process Effect on Mechanical Behavior of PCS-Derived Si-C Ceramics

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Abstract

Strength evaluation of silicon carbide (Si-C) ceramics fabricated from polycarbosilane (PCS) precursor is described. Si-C ceramics was prepared by firing a green body made of the mixture of Si-C nano-powders and a PCS solution at 1273 K in N2 gas for an hour. To obtain dense Si-C, the polymer infiltration and pyrolysis (PIP) process was conducted up to 12 cycles. Si-C ceramics was diced to be rectangle shape, and was subjected to the three-point bending test. Si-C specimens fabricated through PIP processes less than 2 cycles showed non-linear force-displacement curves, whereas those through the processes more than 3 cycles showed linear relations and fractured in a brittle manner. The Young's modulus of 12-cycles-PIPs specimen was 56 GPa, 22-fold of non-PIP specimen. The bending strength was also increased up to 157 MPa with an increase in the number of PIP process. The cause of the influence of PIP process on the mechanical characteristics is discussed using a PCS-derived Si-C model.

Keywords: Polycarbosilane, silicon carbide, mechanical property, strength, polymer infiltration and pyrolysis process.

Introduction

Silicon carbide (Si-C), which is one of the heat resistant ceramics, attracts much attention as a structural material utilized power for microelectromechanical systems (MEMS). Si-C is typically prepared by chemical vapor deposition (CVD) or sintering.¹ Si-C film prepared by CVD is useful for surface modification of a micro device because the material shows superior characteristics, such as small thermal expansion coefficient, less abrasion, high density, and chemical inertness. Si-C structure made by sintering has also good characteristics as does that by CVD. In the cases of CVD and sintering, however, fabricating threedimensional arbitrary shape at the micro scale is very difficult because Si-C basically has excellent mechanical strength and chemical inertness. Several researchers have so far made many efforts to

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produce ceramics MEMS elements by using polymer precursor method.² In this method, a precursor polymer is firstly cast into a micro mold fabricated by UV-thick-photoresist lithography. Then the cast polymer is pyrolyzed in an inert gas in order to convert into self-similar shaped ceramics. However, the polymer-derived ceramics elements are known to become porous, which provides the reliability of ceramics MEMS structures with a negative effect.

We have fabricated polymer-derived Si-C ceramics MEMS made from polycarbosilane (PCS).³ To make dense Si-C ceramics element, the polymer infiltration and pyrolysis (PIP) process was adopted. In this paper, we focus on investigating the mechanical characteristics, such as Young's modulus and bending strength, by means of the three-point bending test. The influence of PIP process on those properties is discussed on the basis of a PCS-derived Si-C model.

Experimental

As the first step for fabricating PCS-derived Si-C ceramics, the 40 %-PCS toluene solution was mixed with Si-C nano-powder derived from PCS to make Si-C powder coated with PCS. The volume ratio of PCS to the powder was 1:10. PCS-coated Si-C powder containing stearic acid of 2 wt.% was filled into a mold under a constant pressure of 80 MPa. Then, the green body was removed from the mold, and fired at 1273 K in Ar gas. After firing, the Si-C body was obtained. Since the body had a lot of pores, it was immersed in a PCS solution to fill it into the pores by means of vacuum degassing. Then, firing at 1273 K for an hour was performed again. The PIP process was conducted multiple cycles (up to 12 cycles) to improve its porosity. The Si-C body was mechanically cut by using a dicing saw to form rectangle-shaped structures measuring 1.0 mm×3.0 mm×0.5 mm, which were utilized as the specimens for the three-point bending test.

Figure 1 shows the relationship between the number of PIP process and porosity. Porosity was defined as a percentage of pore area to cross-section prepared by dicing cut. Porosity was decreased with an increase in the number of PIP process. For



Fig. 1 The number of PIP process vs. porosity.



Fig. 2 Three-point bending test apparatus.

example, the porosity of Si-C ceramics after the first PIP process was approximately 25 %, which decreased to 8 % after 6-cycles-PIPs. Further PIP processes, however, did not bring drastic improvement. After 12-cycles-PIPs, the porosity was 7.5 %, which is only 6.25 % reduction from 6-cycles-PIPs specimen. This is because, in the case of low porosity region, filling a PCS solution into pores was difficult due to very small pore size or formation of closed pores.

Figure 2 shows photographs of three-point bending tester for microscale Si-C ceramics specimen. The tester is able to apply bending force to a specimen in the horizontal direction to easily observe specimen deformation from its overhead with a CCD camera during bending. The load cell and laser displacement meter have a measurement resolution of 0.1 N and 10 nm, respectively. Loading and supporting jigs were made of cemented carbide to restrain elastic deformation of those jigs as small as possible. The distance between supporting points is 2.4 mm. The radius of curvature in supporting and loading jigs is 0.3 mm.

Results and Discussion

Figure 3 shows representative bending forcedeflection relations of Si-C specimens prepared through the PIP processes of 0, 3, 6, and 12 cycles. All the specimens were bent to failure at room temperature. Humidity was not controlled during the test. The loading speed was kept constant to be 0.167 μ m/s. Non-PIP specimen shows non-linear



Fig. 3 Bending force-displacement relations.



Fig. 4 Relationship between the number of PIP process and Young's modulus.

force-displacement relation which slope is very small. The bending force hardly increases even when bending displacement increases until failure. No obvious yield point is seen. 2-cycles-PIPs specimen shows a similar trend as does non-PIP specimen. The maximum bending force is seen at the displacement of 1.5 µm, and then the force gradually drops to failure. At the beginning of bending test for 3-cycles-PIPs specimen, the forcedisplacement relation is linear. The slope is definitely larger than that of non- and 2-cycles-PIPs specimens. The bending force drastically drops at the deflection of 3.2 µm, but the force does not reach to 0 N. By virtue of rapid crack propagation into the specimen, the first fracture occurred, but the entire specimen did not fracture completely. After the first large fracture, probably lots of small fractures happened sequentially at the vicinity of the crack tip. 6- and 12-cycles-PIPs specimens show the same behavior that the force-displacement relation is almost linear until failure. At the deflection of 4.4 µm, bending force immediately drops to 0 N, indicating that brittle failure happened abruptly. From the bending test results described above, it is considered that "apparent" ductilebrittle deformation boundary would exist in Si-C ceramics produced through 2- or 3-cycles-PIPs.



Fig. 5 Weibull distribution of bending strength.

Figure 4 shows the relationship between the number of PIP process and the Young's modulus of Si-C ceramics. The Young's modulus was calculated from the slope of force-displacement relation in the beginning of the test. The modulus of non-PIP specimen is found to be 2.5 GPa on average. This value is very low as with a polymer, which would be caused by low density and insufficiency sintering. With an increase in the number of PIP process to 3 cycles, the modulus remarkably increases to 45 GPa. This indicates that the specimen can rapidly harden by conducting a few PIP processes. After 3-cycles-PIPs processes, the increment in Young's modulus gradually decreases with increasing the number of PIP process. The mean value for 12-cycles-PIPs specimen is 56 GPa, approximately 22 times of that for non-PIP specimen. The maximum value of 68 GPa was obtained, which is about one-sixth of the Si-C bulk value. Compared with PCS-derived Si-C fibers, the value is about 0.36 times. The difference would be caused by poor crystallinity and/or low density of the Si-C body. And also, in the PCSderived Si-C body fired at 1273 K, carbon-rich composition is typically obtained, and it might have given rise to low Young's modulus.

Figure 5 shows the Weibull plot of bending strength for Si-C ceramics. Total 56 specimens were subjected to the bending test. The strength data of all the specimens under respective PIP conditions were lined up on Weibull probability plotting paper, and could be approximated by respective straight lines. This indicates that 2parameter Weibull distribution function is appropriate for the data fitting.⁴ The slopes of respective lines are closely related to the variability of strength for each specimen, and tend towards vertical with an increase in the number of PIP process.

Figure 6 shows the relationships between the number of PIP process, scale, and shape parameters. The closed and open plots are indicative of the scale and shape parameters of Weibull, respectively. The



Fig. 6 Relationship between the number of PIP process and Weibull parameters.

scale parameter of non-PIP specimen is found to be about 9 MPa, which is quite lower than the bulk strength.⁵ The parameter rapidly increases with increasing the number of PIP process as does the Young's modulus. After 6-cycles-PIPs, the parameter drastically increases to 140 MPa, which is approximately 15 times higher than that in non-PIP specimen. The strength increase is probably associated with a decrease in porosity. Multiple PIP processes can definitely reduce the size of pores that is thought to play a role as stress riser, and also can provide the formation of strong PCS-derived Si-C linkages between Si-C particles because of sintering time period accumulation. After 12cycles-PIPs, however, the strength shows another 10 % increase only from the strength of 6-cycles-PIPs specimen. This implies that, in low porosity region after several PIP processes, a change of porosity affecting strength was small. The maximum strength value obtained was 157 MPa, which is only 8.3 % of the strength of Si-C fiber.⁶ On the other hand, the shape parameter of non-PIP specimen is calculated to be 2.6, which increases to 14 with increasing the number of PIP process to 12 cycles. Therefore, the number of PIP process has large influence on the improvement of specimen strength and its scatter.

Figures 7 (a)-(c) show scanning electron microscope (SEM) photographs of the fracture surface of Si-C ceramics produced through the PIP processes of 0, 6, and 12 cycles, respectively. In Fig. 7 (a), non-PIP specimen possesses irregular rough surface indicating that there are a lot of pores inside the body. The fracture surface shows a similar irregularity to side surface, so that fracture origin and river pattern indicating crack propagation direction cannot be found. Meanwhile, as shown in Figs. 7 (b) and (c), the specimen's side surface is relatively smooth, compared with non-PIP specimen. The surface flatness was brought about by conducting multiple PIP processes. These specimens have visible river pattern on their fracture surfaces. The river pattern starts from a



Fig. 7 SEM photographs of fracture surface of PCS-derived Si-C ceramics.



Fig. 8 Schematic of PCS-derived Si-C ceramics specimens before and after multiple PIP processes.

specimen surface and proceeds to the inside. This indicates that the fracture origin would have existed at the vicinity of specimen surface where the maximum tensile stress generated during the bending test.

The mechanism can be explained using the Si-C ceramics model before and after multiple PIP processes, as illustrated in Figure 8. After the first PIP process illustrated in the left figure, PCSderived Si-C nano-powders were coated once with a PCS solution, which was fired once. Although the Si-C body was fired, it was weak. This is because, compared with that of Si-C particles, the rigidity of the coating layer is thought to be very low though the organic precursor was fully pyrolyzed. In addition, because PCS definitely shrunk during the firing, many voids were produced in the coating layer. One weakest PCS linkage between Si-C particles where the maximum tensile stress was produced was firstly broken by applying external force, and then other linkages around the failed link successively fractured one by one even when applied force was small. The successive brittle fractures of pyrolyzed linkages probably appeared as "apparent" ductility in the force-displacement relations. On the other hand, after several PIP processes, as shown in the right figure, the impregnated PCS was fired many times; consequently, many of unnecessary atoms included in the PCS vaporized, and the pyrolyzed PCS layer definitely toughened. By multiple impregnations and firings, the number of voids decreased and those sizes were reduced. Once a crack initiated, it probably propagated soon into strong Si-C body, and finally the entire body would have been fractured.

Conclusions

We fabricated Si-C ceramics micro-elements derived from PCS precursor polymer, and evaluated their mechanical characteristics by means of threepoint bending test. The produced Si-C body was immersed into a PCS solution, and then firing at 1273 was performed. This PIP process was carried out up to 12 cycles, in order to improve their poor porosity. A large number of PIP process yielded PCS-derived Si-C ceramics having better mechanical characteristics.

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Fabrication of PCS-Derived Si-C Ceramics MEMS using Micro Slip Casting Technique



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Background

Power MEMS is one of the significant concerns in the aerospace and energy harvesting fields. In operation of power MEMS device, temperature is typically very high, so that structural materials in the device should possess good heat resistance and mechanical characteristics. Single or poly crystal silicon (SCS/PCS) is commonly used as a structural material in MEMS because MEMS originated from semiconductor industry. SCS and PCS are easily formed with micro or nano scale accuracy, but both materials do not have heat resistance enough to be admitted in power MEMS. In recent years, SiC, one of the heat resistant ceramics, is being used as the structural material in power MEMS. SiC is typically prepared by chemical vacuum deposition or sintering. However, fabricating three-dimensional arbitrary shape of SiC on the micro scale is very difficult due to its superior mechanical strength and chemical inertness. To develop reliable power MEMS device for commercialization, a new fabrication technique for microscale SiC ceramics element should be developed.

Experimental Procedure



Experimental Results & Discussions

The SiC micro gears by slip casting			
The top side of SiC micro gear			
The back side SiC micro gear			
(a) 2 cycles pressurization	(b) 3 cycles pressurization	(c) 6 cycles pressurization	
SiC micro gears fabricated by micro slip casting using WC plate with porosity of 11.1%.			

The shape of produced parts in the number of pressurization cycles during casting was 2, at least, gear. Most of cogs fractured. In the case of 3 cycles pressurization, the shape of gears is better than that prepared in the number of pressurization cycles was 2. The number of cogs surviving increases with an increase in the number of pressurization cycles. After 6 cycles pressurization, all of the cogs are alive, and the MEMS gear was able to be finely fabricated.



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Abstract

A new fabrication technique for silicon carbide (Si-C) microelectromechanical systems (MEMS) is described. Slip casting using UV-thick photoresist SU8 micro mold was carried out for fabrication of three-dimensional Si-C MEMS parts. Ultrahigh molecular weight polycarbosilane (PCS) was used as the precursor. Si-C nano powder was firstly mixed with a PCS solution, and then the slips were cast into SU8 micro mold fabricated on porous tungsten carbide (WC) plate. Firing at 1273 K was conducted for SU8 evaporation and PCS pyrolysis simultaneously. We have succeeded in producing Si-C ceramics micro gears using the "u slip casting" technique. The influences of WC plate porosity and the number of casting cycles on the shape of the produced Si-C MEMS are discussed.

Keywords: Polycarbosilane, silicon carbide, MEMS, slip casting, UV-LIGA.

Introduction

Power MEMS is one of the significant concerns in the aerospace and energy harvesting fields.¹ In operation of power MEMS device, typically temperature rises to several hundred degC, so that the structural materials should possess good heat resistance and mechanical characteristics. Single and poly crystal silicon are used in conventional MEMS devices, but they are not suitable for structural material in power MEMS. In recent years, SiC, which is one of the heat resistant ceramics, is being used as the structural material in power MEMS. SiC is known to have excellent heat resistance, mechanical characteristics, and chemical inertness. However, fabricating three-dimensional arbitrary shape of SiC on the micro scale is very difficult due to its superior mechanical strength and hardness. To develop reliable power MEMS devices, the development of a new fabrication technique for microscale SiC ceramics element is required.

The objective of this work is to propose μ slip casting technique for producing SiC micro elements.

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Fig.1 Construction model of PCS precursor for SiC.

The technique that we have developed is the combination of UV thick-photoresist lithography and conventional slip casting. In this paper, the technique is firstly described. Then, the effects of WC plate porosity and the number of pressurization during casting on the shape of the produced SiC MEMS parts are discussed.

Experimental Technique

Precursor polymer

Polycarbosilane (PCS), which is known as one of ultrahigh molecular weight polymers, is typically being used as a precursor for SiC fiber.² PCS that consists of Si, C, O, and H atoms is combined by heat decomposition of tetramethylsilane. As illustrated in Fig.1, by heating at 1273 K in an inactive gas, PCS is pyrolyzed and changed into SiC ceramics with high ceramics yield.³ In this case, the ceramics yield is theoretically 84 %, and the volume constriction yield is about 1.8 %.⁴ In this work, PCS was employed as a precursor for microscale SiC ceramics parts.

"µ slip casting" technique

Fig. 2 shows a process chart for fabricating SiC ceramics micro parts. At first, SU8 sheet of 50 μ m thick is laminated in five times onto WC plate so as to form a 250 μ m thick SU8 layer. Then UV-thick lithography is performed for fabrication of SU8 micro mold on the plate. The fabrication detail is described in the following section. The slips include SiC nano powder and a PCS solution for 10:1 are



Fig.2 Fabrication process of SiC-MEMS ceramics parts.

mixed in xylene and isopropyl alcohol (IPA). The slips are cast into the mold. During casting, to fill up the mold with the slips, pressurization and depressurization are conducted from the top and back of WC plate, respectively. This process is repeatedly performed up to 12 times in order to investigate the influence of the number of pressurization process on the shape of the cast body. Total amount of slops is 600 μ l, which was kept constant. After the casting, the cast body is fired at 1273 K in N₂ gas for an hour to vaporize unnecessary ingredients in PCS to produce solid SiC. We are able to release the SiC ceramics body after firing because SU8 is dissolved at around 723 K.

SU8 micro mold fabricated on WC plate

As is well known, WC is representative cemented carbide material with a high melting point and high strength. WC plate used for a filter during casting is made by powder sintering, and it includes many pores. We prepared seven kinds of WC plates with different porosity and surface finishing. Fig. 3 representatively shows 11.1%-porosity WC plates with polish and blast surfaces.

SU8 micro mold is fabricated onto those WC plates. Fig. 4 shows a process chart for fabricating SU8 micro mold. It is well known that SU8 is delamination-easy (i.e., easily removable or separable) material from a substrate. In addition, surface roughness and its inhomogeneousness cause expose-light dispersion, which leads to low accuracy patterning. To obtain SU8 micro mold with superior shape and without delamination, fabrication conditions were optimized. The fabrication parameters were employed as follows: pre-bake temperature fixing;368K, post-exposure-



(a) WC plate with polish surface (b) WC plate with blast surface Fig.3 Porous WC plate with the porosity of 11.1%



Fig.4 Fabrication process of SU-8 micro mold.

bake (PEB) temperature fixing;328K and time period, exposure time period, developing temperature and time fixing; 1h period, and laminating temperature.

Results and Discussion

Fabrication of SU8 micro mold

Table 1 lists a summary of SU8 micro mold fabrication on WC plates. The circle, triangle and square plots in the table indicate non-delamination area of more than 90%, 90-50%, and less than 50%, respectively. Higher lamination temperature and longer exposure time period using lower porosity WC plate is found to be better for precisely fabricating SU8 micro mold. Those trends are represented in Fig. 5. As shown in Fig. 5(a), SU8 mold pattern is completely laminated after UV-LIGA process when it was produced through ideal experimental condition. As the condition somewhat deviates from the ideal one, the mold is partly delaminated from WC plate, shown in Fig. 5(b). In the case of the worst condition, SU8 mold is completely delaminated and released from WC plate as shown in Fig. 5(c). Those phenomena are probably related to stress relaxation of SU8 sheet. In this work, the optimum experimental condition



(a) Fine (b) Partly delamination (c) Delamination Fig. 5 Typical SU8 mold fabrication results.

was determined as: laminating temperature, 348K; exposure time period, 75s; PEB time perild,12h.

SiC ceramics MEMS parts

Fig. 6 shows representative examples of the SiC ceramics micro gears produced using µ slip casting technique. All the gears shown in the figure were obtained using WC plate with the porosity of 11.1%. When the number of pressurization cycles during casting was 2, many of cogs are found to fracture. The number of cogs surviving increases with an increase in the number of pressurization cycles. After 6 cycles pressurization, all of the cogs are alive, and the MEMS gear was able to be finely fabricated. The influence of the number of pressurization on on the percentage completion of MEMS gear fabrication is thought to depend on the existence of cracks in the cast body. Any apparent cracks were not seen in the top surface of MEMS gear regardless of the number of pressurization cycles, whereas in the back surface the number of cracks decreased with increasing the pressurization cycles. The number of cracks would likely be related to casting speed and the amount of slips used in one time.

Fig. 7 shows the relationship between the number of pressurization cycles and the crack-tosurface area ratio. The ratio was defined as the ratio of crack portion to surface area of gear, and the crack portion was determined using binary format image method. In the case of 2 pressurization cycles using polished and blasted WC plates with the porosity of 11.1%, the ratio value is found to be 13.3% and 9.7% on average, respectively. Blasted WC plate is able to provide crack-less SiC MEMS parts. This is because surface morphology of the blasted plate was more uniform than that of the polished plate. The uniformity is important for slip casting because the difference in casting speed on the plate causes crack generation. Both the ratio values proportionally decrease with an increase in the number of pressurization cycles. At 6 cycles pressurizations, the crack ratio values for polished and blasted WC plates castings were 6.3% and 2.9%, respectively, which are approximately 7.0% and 6.8% reductions from those at 2 cycles. The reason why many pressurization cycles yielded less cracks in the cast body is probably attributed to the difference in casting speed between small and large SiC particles. During casting, large particles settle down faster than small ones, so that large SiC



Fig. 6 SiC micro gears fabricated by micro slip casting using WC plate with the porosity of 7.9%.



Fig. 7 Relationship between the number of pressurization and the ratio of crack using WC plate with the porosity of 11.1%.

particles exist at around the back surface. This yields large gaps between large particles. The large gaps probably lead to large crack generation. In the case of many pressurization cycles, the thickness of cast layer is thin. So, because the difference in settling down speed between small and large particles is small, small particles can intrude into gaps between large particles; therefore it is difficult to generate large cracks.

Fig. 8 shows the crack ratio as a function of WC plate prosity. Those data indicate the results of 6 cycles pressurization experiments. The crack ratio value at the polished plate porosity of 7.9% is found to be 5.3%, which gradually increases with an increase in the porosity. The largest value was 7.8%, which was brought about using the WC plate of 23.9%-porosity. The increase trend is probably derived from casting speed increase. Using the coarse plates, it would appear that the difference in casting speed between small and large SiC particles differ greatly. In addition, course WC plate makes



Fig. 8 Relationship between WC plate porosity and crack percentage at 6 cycles pressurizations.

its surface morphology rough, so that casting speed would vary from place to place. From those two things, the porosity increase in WC plate is though to yield the number of cracks on its plate. On the other hand, in experiments using blasted WC plates, the crack percentages in surface area of gear stayed constant without respect to porosity. This implies that smaller difference in casting speed from place to place can produce Si-C MEMS parts with lesser cracks. Therefore, the uniformity of surface morphology on WC plate is significant for crackless Si-C MEMS parts.

Conclusions

We proposed " μ slip casting" technique for microscale SiC ceramics parts used for power

MEMS. The technique includes slip casting and UV thick-photoresist lithography. The slips consisted of 100 nm diameter SiC powders and a PCS solution, and were cast into SU8 micro mold fabricated on porous WC plate. We obtained optimum lithography conditions to achieve SU8 micro mold without delamination Using fabricated SU8 micro mold, we showed that larger number of pressurization cycles and the use of low-porosity WC plate were important for SiC ceramics micro parts without large cracks.

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