UNCLASSIFIED

Defense Technical Information Center Compilation Part Notice

ADP012223

TITLE: Innovative And Cost-Effective Microfabrication of Nanoceramic Components

DISTRIBUTION: Approved for public release, distribution unlimited

This paper is part of the following report:

TITLE: Nanophase and Nanocomposite Materials IV held in Boston, Massachusetts on November 26-29, 2001

To order the complete compilation report, use: ADA401575

The component part is provided here to allow users access to individually authored sections of proceedings, annals, symposia, etc. However, the component should be considered within the context of the overall compilation report and not as a stand-alone technical report.

The following component part numbers comprise the compilation report: ADP012174 thru ADP012259

UNCLASSIFIED

V9.7

Balakrishnan G. Nair Ceramatec Inc. Salt Lake City, UT 84119

ABSTRACT

An innovative and cost-effective processing technique has been developed at Ceramatec Inc for the microfabrication of ceramic components requiring very high dimensional tolerance. The materials system is a proprietary nanophase composition called CERCANAM (CERamatec CAstable NAno Material). Scanning electron microscopy revealed that CERCANAM components can be fabricated with dimensional tolerance as high as $\pm 2 \mu m$ for surface features on the die that have dimensions about 1 mm. The process can also be modified to fabricate nanoporous ceramic components with very high surface areas. Components with retained surface areas as high as 67-82% of the starting powder were fabricated. The fabrication process does not involve a high-temperature sintering step, which eliminates the loss of surface area from high temperature sintering. It is anticipated that microcomponents fabricated with specific microstructures and properties will have applications in the optical fiber industry as interconnects, in the electronic packaging industry and the chemical industry.

INTRODUCTION

Conventional micro-machining techniques to form high-precision components (e.g. wet and dry etching) are very slow and expensive processes, and often do not meet the desired production rates and cost criteria required for bulk production of components. Further, most of these processes are specific for silicon, which has a relatively low fracture-toughness (0.7-0.8 MPa.m^{1/2})¹ and is subject to severe corrosion in the high-temperature oxidizing conditions² typical of industrial chemical processes employing microchannel devices. Ceramic materials have excellent corrosion and mechanical properties that make them very attractive for high-temperature applications. However, most current processing techniques for micro-devices made of ceramics are even more expensive than silicon technology. Most of these processes require sintering/pyrolysis at high-temperature that result in at least 10-20% shrinkage.³ Such shrinkage is very difficult to accurately model, and therefore the component dimensions are difficult to control to required tolerances.

MATERIALS AND METHODS

Our process involves a novel casting technique to form net-shape components made of a reaction-bonded nano-ceramic material, which we will refer to henceforth as CERCANAM (for CERamatec CAstable NAno Material). This material was developed as a result of an internally funded research project at Ceramatec, Inc, in Salt Lake City, UT. The specific composition of CERCANAM and the processing technique are considered proprietary and are not relevant to the technical content of this manuscript. The idea of using reaction-bonded nano-ceramics like CERCANAM to fabricate micro components is novel.

Firing temperatures to form the finished component are usually as low as 200-600°C, although it can be as high as 1000°C if necessary. These temperatures are well below sintering temperatures of the ceramic materials that form the primary phase, and therefore there is no loss of dimensional accuracy due to sintering. Figure 1 shows the various processing alternatives for microfabrication of ceramic components using CERCANAM. The ceramic powder, composed either entirely or with a majority phase of sub-micron or nano-sized particulates is mixed with the appropriate reagent and stirred until gellation occurs and water separates out. The gel is collected and a green body is formed by one of the techniques shown in Figure 1, namely slip casting, tape casting, gel casting or extrusion. The casting technique is followed by punching the necessary design using a reusable die that has the "negative" of the required pattern. The die can be fabricated either from conventional materials like silicon using wet or dry etching, or using a photoresist material for use in a lost-mold technique, where the polymer is decomposed or vaporized completely during heat-treatment to form the finished component.⁴





RESULTS AND DISCUSSION

Micro-features on CERCANAM components have been shown to have very high dimensional accuracy with respect to features on the die. Figure 2(a) shows optical micrographs of a copper penny and the impression obtained by the penny on a post-fired CERCANAM specimen (firing temperature: 200° C); figure 2(b) shows SEM micrographs of the same. Digital analysis of the micrographs using image analysis software shows that dimensional accuracies of ± 3 µm over

distances of 2mm have been achieved on the CERCANAM specimen with respect to features on the copper penny. Features as small as 20 µm have been accurately imprinted.



Figure 2 Comparison of features obtained on a post-fired CERCANAM specimen imprinted with a copper penny (right) with respect to features on the penny (left): (a) optical micrographs; (b) SEM micrographs.

Figure 3(b) shows a microfabricated, post-fired CERCANAM component (firing temperature: 600° C) with a design imprinted from an etched silicon wafer with $100 \ \mu m \times 100 \ \mu m$ etch pits, which is shown in figure 3(a). The bases of the pyramids obtained on post-fired CERCANAM specimens have been measured to be $100 \ \mu m \pm 1 \ \mu m$. The attainment of such high dimensional

tolerances illustrates the significant potential of CERCANAM materials for high-precision applications such as optical fiber interconnects, MEMS, etc. However, the fact that the processing technique is very inexpensive relative to other ceramic microfabrication techniques should make it attractive even for other microfabricated components such as microchannel devices and high-power/high temperature electronic packaging, where the attainment of submicron dimensional tolerances are not as critical.





Figure 3 (a) 100 μ m × 100 μ m etch pits on a silicon wafer used as a die for fabricating a slip-cast CERCANAM specimen; (b) pyramids of base dimensions 100 µm \times 100 µm formed on the post-fired CERCANAM specimen. The tolerances on the base of the pyramids are estimated to be $\pm 1 \, \mu m$.

Another advantage of this processing technique is that high-surface area CERCANAM compacts can be obtained by relatively minor changes in the processing technique. The only modification is the addition of a pore-forming phase that can be removed at relatively low temperatures, and the use of a high surface area powder as the primary ceramic phase. Since the material is reaction bonded, and final firing temperatures are well below sintering temperatures, a very high proportion of the surface area (67-82%) of the starting powder can be retained by controlling the

volume fraction of the binding phase through control of processing parameters such as concentration of the reagent and time of reaction. This property of CERCANAM makes it a good choice in certain microchannel devices where a high-surface area ceramic is required as a support for particles of a catalytic or adsorbent phase that can be dispersed in the fine pores of the support.

CONCLUSIONS

Microfabrication of CERCANAM components offers a technologically simple, one-step alternative for complex geometries that would require multiple-step processing with silicon technology. This material/process is expected to have significantly lower processing costs and production times for complex geometries, and can be scaled to large volume output with very high component production rates. Further, CERCANAM materials, due to their inherent thermochemical/thermomechanical stability are attractive for applications where microfabricated components are subjected to high temperatures (600-1000°C) and corrosive environments (e.g high-temperature gas sensing, high-temperature, high-power electronic packaging).

REFERENCES

- 1 Chen, C. P. and M. H. Leipold, "Fracture toughness of silicon," Am. Ceram. Soc. Bull. 59[4] 469-72 (1980).
- 2 Declerck, G. J., "Silicon oxidation," Microelectron. Mater. Processes 164 79-132 (1989).
- 3 L-A. Liew, W. Zhang, L. An, S. Shah, R. Luo, Y. Liu, T. Cross, M. L. Dunn, V. Bright, J. W. Daily and R. Raj, "Ceramic MEMS: New Materials, Innovative Processing and Future Applications," *Am. Ceram. Soc. Bull.* 80 [5] 25-30 (2000).
- 4 U. P. Schonholzer, R. Hummel, Rene and L. J. Gauckler, "Microfabrication of ceramics by filling of photoresist molds," Adv. Mater., **12** [17] 1261-1263 (2000).