§13. Near Net Sintering of Zero Expansion-Pore Free Ceramics by Microwave

Sato, M., Akiyama, R., Takayama, S., Matsubara, A., Iye, M., Akitaya, H.
(National Astronomical Observatory of Japan),
Mastuo, H., Iguchi, M.
(Nihon Ceratech Co., Ltd, Japan)

INTRODUCTION

The large optical mirror has been developed by using ZPF ceramic for the Extremely Large Telescope (ELT) in the field of astronomies [(1)]. NIFS collaborated in the development for microwave sintering of the large mirrors.

ZPF is ceramics that contains Silicon carbide composite (SiC-Si_3N_4) powders as aggregate and of Lithium –alumina -silicate (Li-Al-Si-O) crystallized (glass) powders as matrix. The ceramic has zero expansion and pore free characteristics with high stiffness. The microwave heating has been tried to sinter the segments of this unique ceramics.

In the experiment, it was found that the SiC absorbed microwave energy at low temperature regime. The uniqueness of ZPF ceramics is that the SiC powders works as the heaters distributed uniformly in the heterogeneous compounds at micron scale. The second uniqueness is that the glass matrix began to get energy effectively at higher temperature than 800 degree C. As the result, the glass matrix relayed the role to get microwave energy from silicon carbide. It shows the importance of the material design fitting to the microwave properly that will open the new door to the industries in showing the potentials to replace more metal structural components to ceramics.

2. EXPERIMENTAL

2.1 The ZPF Test Pieces

Sizes of the powders were under 1 micron. They were formed by CIP casting to 122 x122x 20mm tiles and 400 mm round dishes. The Zero-Expansion property is achieved by appropriate mixture of its primal materials in powder with negative thermal expansion (SiC compounds) and that with positive one (Li-Al-SiO) at room temperature. The weight ratio of (SiC) / (Li-Al SiO) was about 60 / 40 . They were degassed in hot air before the sintering and stacked on 2~3 layers in the isothermal blanket and heated in the microwave.

2.2 Heating Behavior under Microwave Irradiation

Fig.1 illustrates the heat curve and the electric power input to the generators. The envelope of the figure showed the microwave. In the low temperature regime from room temperature to 200 degree C, the required power was very small such as 2% duty cycle, i.e. 200 watts. The SiC powders absorbed microwave energy effectively at the low temperature. It increased exponentially to 7.5kw to hold the temperature rising rate of 100 degree C/hour as the dielectric loss factor of SiC decreases when the temperature increases. The rapid increment drastically met the braking point at about 800 degree C. The required microwave power began to decrease exponentially. It downed to 1.25kw at 1220 degree C and jumped up again to the highest level and finished sintering. The mechanism will be explained by activity of Li ions in the Li-Al-Si-O matrix. It is the ternary material and has the eutectic point at 975 degree C. Beyond the temperature, the fraction of liquid phase increased and electricity becomes more active. The discrepancy of the temperature measured by IR (~800) and phase diagram (975) would be explained by the selective heating [(3)]. When the microwave turn on, the hot spots exceed the eutectic point instantly, but in the turn off periods glass diffused the energy to the SiC rapidly. The mechanism works to melt down the work pieces at 1300 degree C in the microwave furnace, but material can be sintered at 1370 degree C in the conventional furnace. The ZPF sintered to the theoretical density at 1270 degree C with the socking time of 15 minute under the atmospheric pressure. The densifications reached to the theoretical value in all the sampling point of the 122 mm (green size) test pieces as. The deformation is very small and the near net sintering achieved to the samples with 100 % densifications.

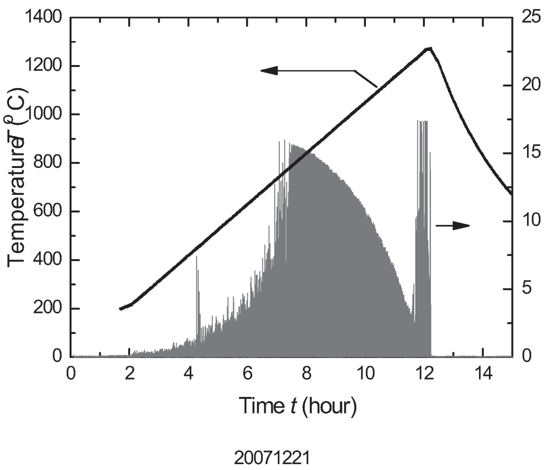

Fig.1 (Upper) Temporal evolution of temperature and electric power to microwave generators

1. H.Akitaya, M. Iye, et el; Application of zero-expansion pore-free ceramics to a mirror of an astronomical telescope, Proc. of SPIE International conference on Astronomical Instrumentation, June 23~28 (2008) Marseille, France