The Research Reports of the Faculty of Engineering, Kagoshima University, No.52 (2010)

Doctoral Dissertation, Graduate School of Science and Engineering, Kagoshima University, Doctorate No.327

Synthesis and Properties of Silicon Nitride–Titanium Nitride Composites by Spark Plasma Sintering

Norhayati AHMAD

1. Introduction

Silicon nitride is difficult to machine via mechanical chip forming processes even with diamond or CBN tools because of their high hardness. A cheaper and more versatile means to shape these materials is EDM. This thesis discussed the preparation of Si_3N_4–TiN composites by Spark Plasma Sintering (SPS) technique and then their physical, mechanical and electrical properties were evaluated for different sintering temperature and correlated with the microstructures. In this thesis, I prepared the composites by using a conventional sintering and in-situ reaction sintering method.

2. Results and Discussions

The fabrication of Si_3N_4–TiN composites by conventional sintering of silicon nitride and titanium nitride powders was examined. SPS was applied to produce electroconductive Si_3N_4-20vol%TiN composites. The sintering mechanism for SPS was a hybrid of dissolution-reprecipitation and viscous flow at 1450 and 1500 ºC and viscous flow at 1600 ºC. At temperatures higher than 1550 ºC, nearly full density (relative density of 98%) was achieved. The Vickers micro-hardness reached a maximum (21.7 GPa) at 1550 ºC. Homogeneous distribution without the agglomeration of TiN particles in the Si_3N_4 matrix was achieved by SPS at 1600 ºC. The composites prepared by SPS at 1550 and 1600 ºC had low electrical resistivity and could be machined by electrical discharge machining. .

The detailed of fabrication of Si_3N_4–TiN composites by in-situ reaction of silicon nitride and titanium powders was examined. Si_3N_4-TiN composites were mixed via planetary ball milling of 70 mass% Si_3N_4 and 30 mass% Ti powders, followed by SPS at 1250-1350 ºC. The sintering mechanism for SPS was a hybrid of dissolution-reprecipitation and viscous flow. The electrical resistivity decreased with increasing sintering temperature up to a minimum at 1250 ºC and then increased with the increasing sintering temperature. The composites prepared by SPS at 1250-1350 ºC could be easily machined by electrical discharge machining.
Composite prepared by SPS at 1300 ºC showed a high hardness (17.78 GPa) and a good machinibility.

Mechanical properties of SPSed Si_3N_4–TiN composites was examined. Composites prepared at 1550 ºC by conventional sintering of silicon nitride and titanium nitride powder had the highest hardness (21.7 GPa) and bending strength (621MPa), while composites prepared at 1300 ºC by in-situ reaction of silicon nitride and titanium powder had the highest fracture toughness (8.39 $MPam^{1/2}$) values. The microstructures and mechanical properties can be tailored by controlling the sintering temperature.

Sintering mechanism of Si_3N_4 based composites by SPS was explained. The mechanism involved during electrical field densification and the properties of silicon nitride–titanium nitride composites are addressed. Liquid phase sintering model has been used in describing the densification mechanism of spark plasma sintering.

3. Conclusions

The fabrications of silicon nitride–titanium nitride composites by Spark Plasma Sintering were established.

Date of Conferment of the Degree: March 25, 2010
University Technology Malaysia