Development of Al_2O_3-Ni FGMs Produced by Spark Plasma Sintering

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Abstract

Ceramic-Metal Functionally Graded Materials (FGM) are of great interest for application as Thermal Barrier Coating (TBC) or Wear Resistant Coating (WRC). Spark Plasma Sintering (SPS) is a promising techniques for time-saving consolidation of laminated/graduated powder systems: SPS is a pressure-assisted electrical sintering method which directly applies a pulsed DC current as heat source. In the present work, production of Al_2O_3-Ni FGMs by means of Spark Plasma Sintering is considered; effect of sintering condition on density, hardness and fracture toughness is studied. Problems correlated to this new processing technology are discussed.

Keywords: FGM, Spark Plasma Sintering, Nickel, Alumina

1. Introduction

Spark Plasma Sintering (SPS) is a new pressure-assisted technology which permits sintering at lower temperatures and shorter times in respect to conventional sintering processes [1]. It is a powerful tool for the production of various type of materials: metals, polymers, ceramics and composite/compounds can be fabricated for various application as electronic materials, structural components, biomaterials etc. Fast sintering at relative low temperature also permits the application of SPS technology in the field of nano-materials; furthermore, peculiarities of SPS make it suitable for production of FGM to be employed as thermal barrier coating, biomaterials, etc [2,3].

Powder to be sintered is stacked in a conductive die (generally made of graphite) and heated by Joule effect using a pulsed DC current. Homogeneous distribution of temperature is important for controlled final properties through the entire volume of the compact. In this work, Nickel-Alumina FGM were considered; problems correlated to production of such materials by Spark Plasma Sintering were analyzed. Various compound with different Nickel-Alumina volume ratio were produced under homogeneous temperature condition. Attained properties were compared to properties attained after Spark Plasma Sintering of a 10mm-thick FGM, constituted by 6 layer: pure Nickel, composite layers with 20%, 40%, 60% and 80% alumina, and pure alumina top layer.

2. Experimental and Results

Alumina powder (A16SG, 0.4 μm) and nickel powder (99.5%, 2.2-3 μm) were used in this investigation; base materials and mixtures were spark plasma sintered to obtain 20mm disk-shaped samples using graphite dies. Different mixtures, in term of volume fraction of alumina, were prepared and named 20%, 40%, 60% and 80%. Metallographic investigation demonstrates that mixing procedure returns good homogeneity (Fig. 1). Base materials were indicated as Nickel (0%) and Alumina (100%). Considering results from a previous work by Casari *et al.* [4] a temperature gradient of 250°C is expected to be attained during spark plasma sintering using graded dies. Limiting condition for spark plasma sintering of Nickel-Alumina composites has to be considered: maximum temperature for sintering nickel in contact with graphite and minimum temperature for good densification of alumina has to be defined.

The Ni-C phase diagram suggests that liquid phase develops at 1326°C at less than 2wt% of carbon: it means that maximum sintering temperature for mixtures containing nickel has to be lower than this critical value. In this way, temperature uniformity during spark plasma sintering has to be taken in account to avoid local melting of nickel in the hottest zone of the compact, i.e. at the contact surface with punches [5]. Spark plasma sintering of alumina was performed at 1300°C, 1350°C and 1400°C: in this conditions, relative density of 94.5%, 97.7% and 99.2% were attained, respectively.

Considering that a temperature gradient of 25°C/mm is expected during spark plasma sintering using graded dies, and that a 10mm-thick sample with 6 layers has to be fabricated, a maximum top layer (100% alumina) temperature of 1350°C was adopted to avoid melting of nickel present in the second layer (80% alumina). In this condition, alumina

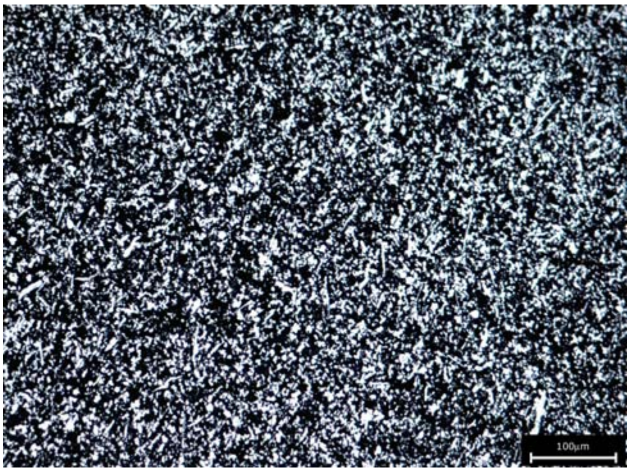

Fig. 1. Homogeneity of microstructure for mixture containing 60% volume fraction alumina (in black)

top layer will experience 1350°C, while, on the other side, a temperature of 1100°C is estimated for the pure nickel layer. Considering a linear temperature gradient, the treatment temperature for 20%, 40%, 60% and 80% alumina layers will be 1150, 1200°C, 1250°C and 1300°C, respectively. On this basis, samples were prepared at these temperature in order to evaluate relative density, hardness, fracture toughness expected for each layer inside the FGM; it has to be pointed out that a maximum temperature of 1200°C was adopted, to avoid local melting due to temperature inhomogeneities during spark plasma sintering [5]. This results in a decrease of relative density and hardness, increasing the volume fraction of alumina, suggesting that 1200°C is to low for sintering alumina.

A layered FGM (10mm-thick, 6 layers) was produced using a graded graphite die, and hardness and fracture toughness of each layer were compared to properties attained after spark plasma sintering of single composites at uniform temperature (Tab. 1). Residual stresses present in the FGM may alter hardness and fracture toughness measurement, but results indicate that the temperature gradient during spark plasma sintering using graded dies is as expected.

3. Summary

Alumina-Nickel composites with different volume ratio were produced by Spark Plasma Sintering and characterized in term of relative density, hardness and fracture toughness. Their properties were compared to those attained after Spark Plasma Sintering of a 6-layers FGM produced under temperature gradient. Results suggest that temperature gradient is effective in production of metal-ceramic FGM.

4. References

[1] M. Tokita, Trends in Advanced SPS Systems and FGM Technology, Proceedings of "NEDO International Symposium on Functionally Graded Materials, Mieparque Tokyo, Tokyo, Japan October 21-22, 1999, pp. 23-33

[2] Trends in Advanced SPS Systems and FGM Technology, Proceedings of "NEDO International Symposium on Functionally Graded Materials, Mieparque Tokyo, Tokyo, Japan October 21-22, 1999

[3] Functionally Graded Materials in the 21st Century: A Workshop on Trends and Forecasts, edited by Kiyoshi Ichikawa, Kluwer Academic Publishers, 2001

[4] F.Casari, M. Zadra, L. Girardini, A. Molinari, Grading of Temperature for Spark Plasma Sintering of Functionally Graded Materials, submitted to Journal of Materials Processing Technology

[5] F.Casari, M. Zadra, L. Girardini, A. Molinari, Evaluation of temperature distribution in Spark Plasma Sintering, submitted to Journal of Materials Processing Technology

Table 1. Properties of single compounds attained after SPS at uniform temperature are compared to those attained after SPS of the FGM sample, under temperature gradient.

Properties	Nickel, 0%	20%	40%	60%	80%	Alumina, 100%
Temperature [°C]	1100	1150	1200	1200	1200	1350
Density %	full density	94.6	88.2	80.9	76.9	97.7
Hardness [HV20]	70±1	243±5	229±9	211±11	155±11	1995±90
K_{IC} [MPa m$^{1/2}$]	-	-	-	-	0.58±0.02	2.08±0.02
FGM Spark Plasma Sintered using graded die						
Expected Temperature [°C]	1100	1150	1200	1250	1300	1350
Hardness [HV20]	80±1	229±4	237±10	355±14	812±42	1893±32
K_{IC} [MPa m$^{1/2}$]	-	-	-	-	1.35±0.02	1.86±0.02