EXPERIMENTAL ANALYSIS AND PRESSURELESS SINTERING OF POWDER PROCESSED FUNCTIONALLY GRADED NICKEL ALUMINA PLATES

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Bahan yang disusun secara berfungsi (*functionally graded material* – FGM) adalah salah satu kelas dari bahan-bahan termaju yang direka khusus bagi menangani isu kesukaran menggabungkan bahan-bahan berlainan yang mempunyai ciri-ciri berbeza serta untuk menyelesaikan masalah percantuman antara zarah dengan mengurangkan kepekatan tekanan antara lapisan bersebelahan. Sistem FGM nikel-alumina (Ni/Al_2O_3) dikenali sebagai bahan tahan haba dan stabil pada suhu tinggi (melebihi 1000ºC). Di dalam kajian ini, sampel FGM multi lapisan Ni/Al_2O_3 telah dibentuk melalui prosedur serbuk metalurgi dan *sintering* tanpa tekanan, kemudian dipercirikan sifat fizikal dan mikrostruktural. Metodologi penyelidikan adalah termasuk kajian awalan numerikal menggunakan kaedah unsur terhingga (ANSYS) melibatkan plat silinder FGM Ni/Al_2O_3. Dalam prosedur eksperimental, sampel-sampel silinder bagi FGM Ni/Al_2O_3 6 dan 9 lapisan telah dibuat melalui kaedah serbuk metalurgi, dipadatkan pada tekanan ekapaksi 414 MPa diikuti dengan proses *sintering* tanpa tekanan pada suhu 1200ºC dalam persekitaran argon berdasarkan parameter pemprosesan yang berbeza. Komposisi setiap 4 dan 7 lapisan-lapisan perantaraan yang disusun masing-masing mempunyai peratusan berat nikel 80, 60, 40 dan 20 wt.% dan 85, 70, 60, 50, 30, 20 dan 10 wt.%. Untuk setiap sampel FGM, masa *sintering* telah divariasikan (3 jam dan 4 jam) untuk membandingkan sifat fizikal dan evolusi mikrostruktur sampel yang dibuat. Sampel yang di*sinter* selama 4 jam menunjukkan sifat yang lebih baik dari segi kepadatan relatif (masing-masing 57.64% dan 62.06% bagi sampel berlapis 6 dan 9) dan keliangan yang berkurang (42.36% untuk 6 lapisan berlapis dan 37.94% untuk 9 lapisan berlapis). Tingkah laku pengecutan dalam semua sampel meningkat dengan peningkatan komposisi seramik Al_2O_3, sementara pengecutan jejari sampel berlapis 6 menunjukkan lebih banyak perubahan berbanding dengan sampel berlapis 9 kerana perbezaan yang lebih besar dalam pengembangan haba yang terhasil antara lapisan. Walaubagaimanapun, keputusan ujian kekerasan mikro tidak berada pada tahap yang memuaskan seperti yang dijangka memandangkan nilai kekerasan menurun walaupun komposisi seramik meningkat di setiap lapisan. Ini disebabkan proses transformasi serbuk alumina menjadi seramik keras adalah tidak lengkap dan peningkatan rongga-rongga keliangan dalam struktur lapisan adalah selari dengan peningkatan kandungan seramik. Mikrograf-mikrograf optik menunjukkan bahawa taburan zarah nikel dan alumina dalam setiap lapisan adalah seragam serta garisan muka antara setiap lapisan adalah selari, justeru mengisyaratkan proses persebatian dan menekan yang baik. Bukti keseragaman campuran setiap lapisan turut disokong oleh analisis spektroskopi penyebaran tenaga sinar-x (EDS) yang menunjukkan peratusan nikel dan alumina dalam komposisi yang sesuai untuk setiap lapisan. Mikrograf-mikrograf optik turut mempamerkan ciri-ciri peralihan mikrostruktur yang beransur-ansur daripada lapisan logam (Ni) tulen kepada seramik (Al_2O_3) tulen mengikut pola pertambahan dalam peratusan komposisi seramik bermula dari struktur mikrostruktur jenis seramik bertaburan, diikuti oleh jenis berangkaian dan seterusnya struktur zarah-zarah logam di dalam matriks seramik. Mikrograf-mikrograf pengimbas mikroskop elektron (SEM) mendedahkan kewujudan liang mikro dan retak mikro terutamanya dalam struktur yang tinggi kandungan alumina. Walaubagaimanapun, dalam beberapa sampel yang diperhatikan, didapati bahawa kewujudan liang adalah kurang dalam struktur sampel yang di*sinter* selama 4 jam berbanding yang di*sinter* selama 3 jam. Oleh itu masa *sintering* selama 4 jam didapati dapat membenarkan lebih banyak proses penggabungan dan pengikatan antara partikel berlaku.

ABSTRACT

Functionally graded material (FGM) is a class of advanced materials innovated particularly to address the issue of integrating different materials of dissimilar properties and resolve the inter-particle bonding problems by reducing the stress concentration between adjacent layers. Nickel-alumina (Ni/Al_2O_3) FGM system is famously recognized as heat resisting materials and stable at high temperature (above 1000ºC). In this research, multilayered Ni/Al_2O_3 FGM samples were designed and fabricated through powder metallurgy and pressureless sintering procedures and then were characterized in terms of their physical and microstructural properties. The research methodologies include preliminary numerical studies using finite element method (ANSYS) involving cylindrical plate of Ni/Al_2O_3 FGM. In the experimental procedures, cylindrical samples of 6 and 9 layered Ni/Al_2O_3 FGM samples were fabricated through powder metallurgy route, compacted at 414 MPa uniaxial pressure followed by pressureless sintering process at 1200ºC in argon atmosphere based on different processing parameters. The composition of each 4 and 7 graded interlayers, with respect to the Ni wt.% are 80, 60, 40 and 20 wt.% and 85, 70, 60, 50, 30, 20 and 10 wt.%, respectively. For each FGM sample, the sintering times were varied (3 hours and 4 hours) to compare the physical properties and microstructural evolution of the fabricated samples. Samples which were sintered for 4 hours showed better properties in terms of relative densities (57.64% and 62.06% respectively for 6 and 9 layered samples) and reduced porosities (42.36% for 6 layered samples and 37.94% for 9 layered samples). Shrinkage behavior in all samples increased as the composition of ceramic Al_2O_3 increase, while radial shrinkage of 6 layered samples showed more changes compared to 9 layered samples due to larger differences in resultant thermal expansion between layers. Nevertheless, the microhardness results were not in satisfactory level as expected and the hardness values were declining although the ceramic composition was increased in each layer. This is due to the incomplete transformation of alumina into hard ceramic and increasing of porosities in the graded structure as the ceramic content rises. Optical micrographs revealed uniform distribution of nickel and alumina particles in each layer as well as parallel interface which confirmed proper blending and pressing processes. The uniformities were supported by the Energy Dispersive X-Ray Spectroscopy (EDS) analyses. The optical micrographs also exhibited gradual microstructural transition characteristics from pure metal to pure ceramic with the addition in the ceramic fraction from a ceramic-dispersed type structure, followed by a network type and subsequently alternate dispersive structure of metallic phase in the ceramic matrix. Micrographs of Scanning Electron Microscopy (SEM) analyses further revealed the presences of micro pores and micro cracks especially in the alumina-rich structures. However, in some samples it was evident that there were fewer pores observed in the structure of samples sintered for 4 hours sintering time compared to those of 3 hours. Therefore it is suggested that 4 hours sintering time will allow more inter-particle connectivity and bonding to take place.

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LIST OF SYMBOLS

HV	Vickers Hardness
MgO	Magnesium oxide
TiO_2	Titanium dioxide
α	Coefficient of thermal expansion (CTE)
E	Elastic modulus
υ	Poisson's ratio
ρ	Density
ε_T	Thermal strain
σ_T	Thermal stress
ΔT	Temperature change
ℓ_g, ℓ_s	Initial green compact length, Sintered length

LIST OF ABBREVIATIONS

AP	Archimedes Principle
APS	Air Plasma Spraying
CAP	Cathodic Arc Plasma Ion Plating
CLC	Classical Laminated Composite
CVD	Chemical Vapour Deposition
DC	Direct Current
D-gun	Detonation Gun Spraying
EDS/EDX	Energy Dispersive X-Ray Spectroscopy
EPD	Electrophoretic Deposition
FEA	Finite Element Analysis
FEM	Finite Element Method
FEM	Finite Element Modelling
FG	Functionally graded
FGM	Functionally graded material
FRP	Fibre-reinforced polymers
HIP	Hot Isostatic Pressing
HVAS	High Velocity Arc Spraying
HVFS	High Velocity Flame Spray
HVOF	High Velocity Oxy-fuel Spraying
IPM	Image Processing of MATLAB
KJMA	Kolmogorov–Johnson–Mehl–Avrami
LROM	Linear Rule of Mixture
LVOF	Low Velocity Oxy-fuel Spraying
MROM	Modified Rules of Mixtures
PC	Pulsed Current
PED	Pulse Electrodeposition
PM	Powder Metallurgy
PRC	Pulsed Reversed Current
PS	Pressureless Sintering
PVD	Physical Vapour Deposition
ROM	Rule of Mixture

SPS	Spark Plasma Sintering
TBC	Thermal barrier coating
VPS	Vacuum Plasma Spraying
vol.	Volume
wt.	Weight

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