UNIVERSITI PUTRA MALAYSIA

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MEHRAN MASOUDI

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DOCTOR OF PHILOSOPHY

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By

MEHRAN MASOUDI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia, in fulfillment of the requirement for the degree of Doctor of Philosophy

SYNTHESIS AND CHARACTERIZATION OF Ni-Al_2O_3-Cr, Ni-Al_2O_3-SiC AND Ni-SiC-Cr NANO HYBRID COMPOSITES

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Chairman: **Associate Professor. Mansor Hashim, PhD**

Faculty: **Science**

This research is focused on the preparation of nickel base composites having nano particles of chromium, alumina and silicon carbide in the form of mono and hybrid composites by using a co-electrodeposition technique in nano powders of SiC, Cr, and Al_2O_3 with a 50 nm mean dimension were introduced into the conventional nickel plating Watt's bath. The prepared new hybrid composites, mono nickel based composites and pure nickel layers were subjected to different tests to characterize their surface morphology, crystalline structure, mechanical properties and hot oxidation resistance. The microstructure and composition of the composite films were studied with field emission scanning electron microscopy (FE-SEM), XRD spectroscopy and EDS techniques. Micro hardness and wear tests were carried out on coated samples to investigate the mechanical properties.

The test results showed effectiveness and flexibility of the co-electrodeposition technique in creating mono and more complicated nickel base composite layers. The novel Ni-Al_2O_3-Cr composite layer showed considerable improvement in hot oxidation resistance compared to the pure nickel layer and the Ni-Al_2O_3 composite; and it enhanced mechanical properties compared to Ni and Ni-Cr composite films. This newly created hybrid composite could be a good substitution for commercial Ni-Al_2O_3 and Ni-Cr composites.

The fabricated novel Ni-Al_2O_3-SiC composite film showed superior mechanical properties among all the mono and hybrid composites studied in this research; and, by comparison with the Ni-Al_2O_3 composite, exhibited enhanced hot oxidation resistance. Its high hardness and wear resistance along with acceptable thermal stability makes the Ni-Al_2O_3-SiC composite film a good choice for coating many industrial parts and components as an anti-oxidation and anti-wear protective film.

The newly fabricated Ni-SiC-Cr nanocomposite coating displayed the highest hot oxidation resistance among all the tested composites in this study owing to chromium's excellent corrosion and oxidation resistant properties. Advantageously, it formed silicon oxide that reduced the diffusion rate at elevated temperatures. The SiC hard and anti-wear particles rendered good mechanical properties to the synthesized hybrid composite. The altered surface morphology, fine crystalline structure, advanced oxidation resistance and improved mechanical properties enable Ni-SiC-Cr composite films to protect metallic parts in high severe corrosive, abrasive and thermal working conditions.

A quantitative technique for measuring grain boundary volume percentage using XRD test results have been suggested and tested successfully in this thesis. This

technique could be a reliable base for future fundamental studies on nano materials deformation mechanisms.

The achievements of the project work laid out in detail in this thesis can be summarized in the following points:

- Successful co-electrodeposition of the Ni- Al_2O_3, Ni-SiC and Ni-Cr mono composites by an electroplating technique.
- Preparation of novel Ni-Al_2O_3-Cr hybrid nanocomposite coatings with excellent mechanical properties, improved anti-oxidation by 55% and attribute advanced thermal resistance.
- Preparation of novel Ni-Al_2O_3-SiC hybrid nanocomposite coatings with superior mechanical properties (three times harder than pure nickel film) and improved hot oxidation resistance.
- Preparation of novel Ni-SiC-Cr hybrid nanocomposite coatings having superior hot oxidation resistance (90% better than pure nickel film) and improved mechanical properties especially wear resistant behaviour.
- Detailed study and discussion on the surface morphology, crystalline structure and texture of the created mono and hybrid composite layers.
- Detailed study comparative on mechanical characterizations of a pure nickel film and mono as well as hybrid composite coatings.
- Detailed study on the oxidation kinetics of prepared MMC coated samples having different varieties in the form of Cr, SiC and Al_2O_3 filler substances.
- Formulation of a new quantitative technique for measuring the grain boundary volume percentage in polycrystalline materials.

Abstrak Tesis yang dikemukakan kepada Senat Universiti Putra Malaysia

Sebagai memenuhi keperluan untuk ijazah Doktor Falsafeh

SINTESIS DAN PENCIRIAN KOMPOSIT HIBRID NANO Ni-Al_2O_3-Cr, Ni-Al_2O_3-SiC DAN NI-SiC-Cr

Oleh

MEHRAN MASOUDI

January 2013

Pengerusi : Associate Professor Mansor Hashim, PhD

Fakulti : Sains

Fokus tesis ini adalah mengenai penyediaan komposit asas nikel yang mempunyai partikel nano kromium, alumina dan silikon karbida dalam bentuk komposit mono dan komposit hibrid dengan menggunakan teknik pemendapan elektro bersama elektro. Serbuk nano SiC, Cr, dan Al_2O_3 dengan min dimensi 50 nm telah diperkenalkan kepada saduran nikel secara konvensional iaitu Watt's bath. Komposit hibrid baru yang telah disediakan, komposit berasaskan nikel mono dan lapisan nikel tulen telah melalui ujian yang berbeza untuk mengenalpasti ciri-ciri morfologi permukaan, struktur hablur, sifat mekanik dan rintangan pengoksidaan panas masing-masing. Mikrostruktur dan komposisi komposit filem telah dikaji dengan pancaran mikroskopi elektron imbasan (FE-SEM), spektroskopi XRD dan teknik EDS. Ujian kekerasan mikro dan ujian kehausan telah dijalankan ke atas sampel bersalut untuk menyiasat sifat-sifat mekanik.

Keputusan ujian menunjukkan keberkesanan dan fleksibiliti teknik pemendapan elektro bersama dalam mewujudkan lapisan komposit mono dan lapisan komposit berasaskan nikel yang lebih rumit. Lapisan komposit Ni-Al_2O_3-Cr yang baru menunjukkan peningkatan rintangan pengoksidaan panas berbanding dengan lapisan nikel tulen dan Ni-Al_2O_3 malah sifat-sifat mekanik komposit telah dipertingkatkan berbanding komposit filem Ni dan Ni-Cr. Komposit hibrid yang diperkenalkan ini boleh menjadi pengganti baik untuk komposit Ni-Al_2O_3 dan Ni-Cr komersial.

Fabrikasi komposit filem Ni-Al_2O_3-SiC yang baru menunjukkan sifat-sifat mekanik unggul antara semua mono dan deposit hibrid yang telah dikaji dalam penyelidikan ini dan dalam perbandingan dengan komposit Ni-Al_2O_3, menggambarkan peningkatan rintangan terhadap pengoksidaan panas. Komposit filem Ni-Al_2O_3-SiC dengan kekerasan dan rintangan haus yang tinggi bersama-sama dengan kestabilan terma yang bersesuaian membuatkan Ni-Al_2O_3-SiC menjadi pilihan komposit filem yang baik untuk saduran banyak bahagian dan komponen industri sebagai lapisan anti pengoksidaan dan lapisan pelindung anti-kehausan.

Fabrikasi baru salutan komposit nano Ni-SiC-Cr menunjukkan rintangan pengoksidaan panas tertinggi antara semua komposit yang diuji dalam kajian ini disebabkan oleh tahap tahan kakisan kromium yang sangat baik dan sifat tahan pengoksidaan serta peranan silikon oksida yang terbentuk dalam mengurangkan kadar respan pada suhu tinggi. Partikel keras dan anti-haus SiC memberikan sifat-sifat mekanik yang baik kepada komposit hibrid disintesis ini. Morfologi permukaan yang telah berubah, struktur kristal yang halus, rintangan pengoksidaan yang ditingkatkan dan sifat-sifat mekanik yang bertambah baik membolehkan komposit filem Ni-SiC-Cr melindungi bahagian-bahagian logam ketika berada dalam keadaan pengakisan, penglelasan dan kerja panas yang teruk.

Teknik kuantitatif bagi mengukur jumlah peratusan isipadu sempadan butiran menggunakan keputusan ujian XRD telah dicadangkan dan diuji dengan jayanya dalam tesis ini. Teknik ini boleh menjadi asas yang boleh dipercayai untuk kajian asas mekanisme canggaan bahan nano.

Pencapaian kerja projek yang dibentangkan secara terperinci dalam tesis ini boleh diringkaskan melalui perkara-perkara berikut:

- Kejayaan pemendapan elektro bersama komposit mono Ni-Al_2O_3, Ni-SiC dan Ni-Cr melalui teknik penyaduran elektro.
- Penyediaan salutan komposit nano hibrid Ni-Al_2O_3-Cr baru dengan sifat mekanikal yang diperbaiki, sifat anti-pengoksidaan yang lebih baik dan rintangan haba yang meningkat sebanyak 50%.
- Penyediaan salutan komposit nano hibrid Ni-Al_2O_3-SiC baru dengan sifat mekanikal unggul (tiga kali lebih kuat daripada filem nikel tulen) dan rintangan pengoksidaan panas yang lebih baik.
- Penyediaan salutan komposit nano hibrid Ni-SiC-Cr baru g mempunyai rintangan pengoksidaan panas yang mantap (90% lebih baik daripada saduran nikel tulen) dan sifat-sifat mekanik yang bertambah baik terutama ciri kehausan.
- Kajian terperinci dan perbincangan morfologi permukaan, struktur kristal dan tekstur mono yang telah dihasilkan serta lapisan komposit hibrid.
- Kajian perbandingan terperinci ciri mekanikal filem nikel tulen, serta salutan komposit mono dan hibrid secara perbandingan.
- Mengkaji kinetik pengoksidaan sampel bersalut MMC yang disediakan dengan pelbagai bahan pengisi Cr, SiC dan Al_2O_3 yang berbeza.
- Penghasilan teknik kuantitatif baru bagi mengukur jumlah peratusan isipadu sempadan butiran dalam bahan polihablur.

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Lastly and most importantly, I wish to thank my colleagues who become my best friends in the magnetic materials research group. I am pleased about your hospitality, opinions and highly support and it was an honour to work and be with you all.

APPROVAL

I certify that a Thesis Examination Committee has met on ---------- to conduct the final examination of Mehran Masoudi on his PhD thesis entitled "**Synthesis and Characterization of Ni-Al_2O_3-Cr, Ni-Al_2O_3-SiC and Ni-SiC-Cr Nano Hybrid Composites**"in accordance with the Universities AND University Colleagues Act 1971 and the Constitution of the Universiti [P.U.(A)106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy degree.

Members of the Thesis Examination Committee were as follows:

Chairperson, PhD
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Name of Faculty
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(Chairperson)

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

MEHRAN MASOUDI

Date: 10st January 2013

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