SYNTHESIS, CRYSTALLIZATION KINETIC AND OPTICAL PROPERTIES OF EUROPIUM DOPED TELLURITE NANO - GLASS

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This thesis is specially dedicated to:

My beloved parents,
Sazali Idris, Zaini Zainuddin

My supportive siblings,
Addam, Lilli, Dianna, Djohan

My dedicated lecturers,

My endless spirits

and all my friends.

.....thanks.....

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ABSTRACT

Tellurite glass of composition (80-x) TeO_2 – 5 Na_2O – 15 MgO – (x) Eu_2O_3, with the concentration region of $0 \leq x \leq 2.5$ mol% was prepared using conventional melt-quenching technique. The transition temperature, T_g, crystallization temperature, T_c and melting temperature, T_m were determined using Differential Thermal Analysis (DTA). The glass with nano-crystalline particle was prepared by heating the as-cast glass at temperature 15 - 20 ^oC above the T_c. The X-Ray Diffraction (XRD) technique with the Scherrer equation was used to determine the size of nano- crystalline particle of the samples while the Scanning Electron Microscopy (SEM) technique was used to identify the formation of nano-crystalline phase. The crystallization kinetics was investigated by using DTA at various heating rate while the glass density and hardness was determined by Precisa Densitometer and Vickers microhardness, respectively. Meanwhile, the emission characteristic was evaluated using the Photoluminescence Spectroscopy (PL). The thermal analysis showed that the T_g and T_c increased with the increasing of Eu^{3+} content while T_m showed a break in linearity as the Eu^{3+} increased. The result also showed that the glass stability up to 109.29. XRD spectra confirmed the presence of nano particles with an average diameter around 68.7 nm. Meanwhile the SEM studies revealed the existence nano-crystalline morphology which was associated with the existence of crystallized phase. The influence of Eu^{3+} content on crystallization kinetics showed a shift of crystallization temperature peak towards a higher temperature with the increasing of heating rate as described by Kissinger and Ozawa method. The activation energy (E_a) was found to decrease from 306.9 eV to 48.9 eV with an increasing of dopant concentration. The density was found to be in the range of 5.234 to 5.334 g cm^{-3} while the Vickers microhardness was found to vary from 2.59 to 2.84 GPa depending on the dopant concentration. A detailed study on the luminescence spectra showed that all emission peaks for $^5D_0 \rightarrow {}^7F_0$, $^5D_0 \rightarrow {}^7F_1$, $^5D_0 \rightarrow {}^7F_2$, $^5D_0 \rightarrow {}^7F_3$ and $^5D_0 \rightarrow {}^7F_4$ transitions were found to be around 568 nm, 600 nm, 628 nm, 664 nm and 712 nm, respectively. It was also found that the heat-treated glass and the inverse quality factor decreased with increasing Eu^{3+} dopant concentration.

ABSTRAK

Kaca tellurite dengan komposisi (80-x) TeO_2 – 5 Na_2O – 15 MgO – (x) Eu_2O_3, mempunyai kepekatan $0 \leq x \leq 2.5$ mol% telah disediakan menggunakan teknik pelindapan leburan konvensional. Suhu peralihan, T_g, suhu penghabluran, T_c dan suhu lebur, T_m ditentukan menggunakan analisis kebezaan terma (DTA). Kaca dengan hablur nano diperolehi dengan memanaskan kaca pada suhu 15 - 20 ^oC di atas suhu penghabluran, T_c. Teknik pembelauan sinar-X (XRD) dan persamaan Scherrer digunakan bagi menentukan saiz zarah nano hablur manakala teknik pengimbas mikroskopi elektron (SEM) digunakan bagi mengenal pasti pembentukan fasa nano hablur. Kinetik penghabluran dikaji dengan menggunakan DTA pada kadar pemanasan yang berbeza manakala ketumpatan dan kekerasan kaca masing-masing ditentukan dengan menggunakan Precisa Densitometer dan kekerasan mikro Vickers. Sementara itu, pancaran dinilai dengan menggunakan spektroskopi kefotopendarcayaan (PL). Untuk analisis terma, T_g dan T_c meningkat dengan peningkatan Eu^{3+} sementara T_m tidak menunjukkan peningkatan linear denagn penambahan kepekatan Eu^{3+}. Keputusan juga menunjukkan bahawa julat kestabilan kaca sehingga 109.29 ^oC boleh dicapai. Spektrum XRD membuktikan kewujudan zarah hablur nano dengan saiz purata diameter 68.7 nm. Sementara itu, kajian SEM membuktikan morfologi hablur nano yang boleh dikaitkan dengan kewujudan fasa hablur. Pengaruh kandungan Eu^{3+} pada kinetik penghabluran menunjukkan peralihan suhu puncak penghabluran ke arah yang lebih tinggi dengan peningkatan kadar pemanasan seperti diterangkan menggunakan kaedah Kissinger dan Ozawa. Tenaga pengaktifan (E_a) didapati menurun daripada 306.9 eV hingga 48.9 eV dengan peningkatan kepekatan pendopan. Ketumpatan kaca didapati berada dalam julat 5.234 hingga 5.334 g cm^{-3} manakala kekerasan mikro Vickers dari 2.59 hingga 2.84 GPa bertambah mengikut kepekatan pendop. Kajian yang teliti ke atas spektrum pendarcayaan menunjukkan bahawa semua pancaran bagi transisi $^5D_0 \rightarrow {}^7F_0$ $^5D_0 \rightarrow {}^7F_1$, $^5D_0 \rightarrow {}^7F_2$, $^5D_0 \rightarrow {}_7F_3$ dan $^5D_0 \rightarrow {}_7F_4$ masing-masing didapati berada di sekitar 568 nm, 600 nm, 628 nm , 664 nm dan 712 nm. Didapati juga bagi kaca yang dirawat haba, faktor kualiti songsang menurun dengan pertambahan kepekatan Eu^{3+}.