

Synthesis and microstructural study of 50AgI-50[0.5Ag₂O+0.25MoO₃+0.25P₂O₅] glass-ceramic composites

*¹Shivaprakash Y, ²Satish M, ³Dinesh C.M

^{*1}Department of Physics, Govt. First Grade College, Devanahalli-562110

²Department of Physics, Govt. First Grade College, Doddaballapura - 561203

³Department of Physics, Govt. First Grade College, Chinthamani - 563125

Abstract

The precursor glass system 50AgI-50[0.5Ag₂O+0.25MoO₃+0.25P₂O₅] has been prepared by melt quenching technique and they are converted to glass-ceramic composites by simple heat treatment technique at 100°C at 2h, 4h, and 8h. The X-ray diffraction and scanning electron microscopy have been performed on the samples. The analyses show that alteration in the grain size and grain boundaries at different heat treatment time. The particle size of 1 micron appear when the samples are heat-treated at maximum time of 8h.

Key words: Glass-ceramics, Silver glasses, Microstructure, Annealing technique, Quenching technique, XRD, SEM.

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I. INTRODUCTION

Glass-ceramic (GC) composites are finding more demand in the fields such as electronics, communication, solid state battery technology, solar energy technology etc. due to their interesting and more flexible properties. AgI-Ag₂O-P₂O₅ is well known to form glass over a wide range of compositions¹. Further it is known that in this glass Ag⁺ ions are the main charge carriers and the conductivity values fall in the range of 10⁻⁴-10⁻¹ S/cm and hence they are known as fast ion conductors. This glass system forms a three dimensional extended network of phosphate groups and Ag⁺ ions occupy the interstitial position^[2-4]. Recently it has been reported that the conditional glass formers such as, MoO₃, V₂O₅ etc. increases the glass forming ability and also improve the physical properties such as conductivity, elastic, optical^[5]. Controlled heat-treatment of such glasses also improves the physical properties, for example the formation of α-AgI stabilized composites in AgI-Ag₂O-MoO₃ glass system leading to enhancement in the conductivity^[6]. In this communication we report the synthesis and morphological characterization of 50AgI-50[0.5Ag₂O+0.25MoO₃+0.25P₂O₅] (AAMP) glass-ceramic composites. These glass-ceramics are expected to have higher conductivity than the post glass.

II. EXPERIMENTAL

The basic glass with composition 50AgI-50[0.5Ag₂O+0.25MoO₃+0.25P₂O₅] was prepared by melt quenching method. The starting materials, AgI, AgNO₃, MoO₃ and P₂O₅ were taken in appropriate proportions and mixed thoroughly in a porcelain crucible, then heated in an electrical furnace in the open atmosphere.

The nitrogen gas was went out at 250°C, then temperature of the furnace was raised gradually till the mixture melts completely. Thus obtained homogenous melt was quenched at the temperature around 800°C. The flat random pieces were obtained by pouring the melt on a brass plate and immediately pressed by another brass plate. The samples were deep brown in color. These glasses were heat treated under controlled temperature in three steps (Room temp-> 100°C for 2 to 8h -> allowed to cool down to Room temp) to transform the basic glasses into glass-ceramic composites. The XRD analysis was carried out using the fine powder of the samples to check the amorphous/crystalline/partially crystalline nature. The flat pieces of the samples were examined thoroughly using SEM to study the morphological changes.

III. RESULTS AND DISCUSSION:

The glass-ceramic composites are partially crystalline materials consist of randomly oriented, fine-grained crystallites in a residual glass matrix formed by the controlled heat treatment technique^[7]. The fine powders and flat pieces of AAMP samples heat-treated at various length of time (2h-8h at 100°C) were used for XRD and SEM analysis. The XRD analysis was carried out using the powder samples and thus obtained pattern is shown in Fig1. The gradual crystallization peaks are seen only for S3 and S4 samples, indicating that the

samples are transformed from complete glass to partially crystallized glass-ceramics after 4 h of heat treatment at 100°C. The crystalline phases were assigned to be AgI (*Hexagonal, Primitive, 2θ=39.14°) based on JCPDS cards (Id No.832 043).

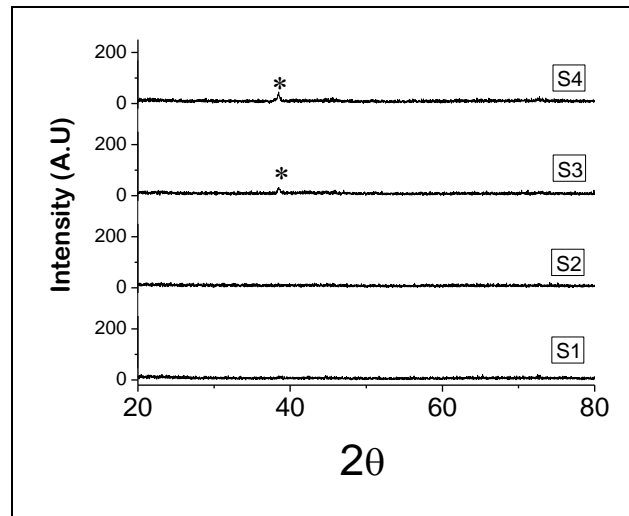


Figure1: XRD plots of AAMP samples. S1- Base glass without heat treatment, S2- GC heat-treated at 100°C for 2h, S3- GC heat-treated at 100°C for 4h and S4 - GC heat-treated at 100°C for 8h. * Crystallite Phase AgI (2θ=39.14°)

The SEM images of the flat pieces heat-treated at various time is shown in Fig 2, which clearly show the alteration in the grain size and grain boundaries. In slide (a) only homogenous glass-phase can be seen. With subsequent heat-treatment for 2, 4 and 8 h, one can clearly see in the slides (c)-(d) that, phase begin to grow gradually. At maximum heat treatment time of 8 h, the particles of size ~1µm are clearly visualized. The XRD and SEM results seem to be consistent and showing the partial crystallization of AgI phase.

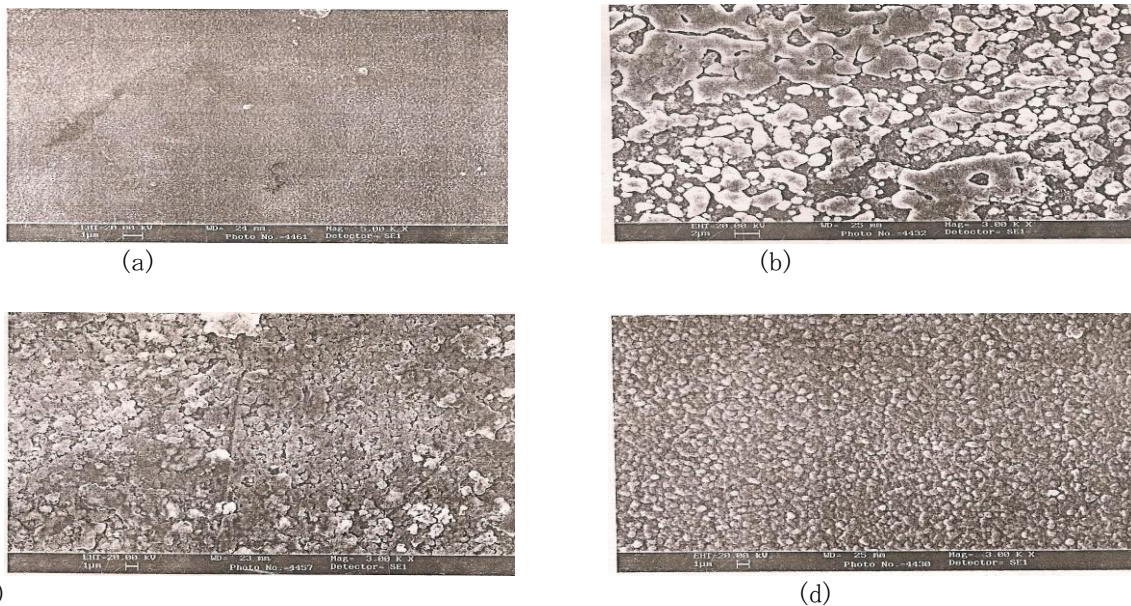


Figure2: SEM records of AAMP Glass-ceramics. (a) Base glass without heat treatment, b) GC heat-treated at 100°C for 2h, (c) GC heat-treated at 100°C for 4h and (d) GC heat-treated at 100°C for 8h.

IV. CONCLUSIONS

The AAMP glass-ceramic composites have been synthesized by heat treatment technique. These glass-ceramics are expected to give improved elastic and electrical properties. Morphological characterization by XRD and SEM show that there is alteration of grain size and grain boundaries due to different length of heat-

treatment time. For maximum heat-treatment time of 8h, the particle sizes of 1µm are seen. These glass-ceramics, which is likely to be in the AgI phase, are expected to give improved electrical conductivity compared to the post glass.

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