**Acknowledgements**

The authors would like to acknowledge the support of the University of Connecticut Research Foundation.

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*Received and accepted*
*9 November 1973*

Y. V. V. R. S. MURTY
T. Z. KATTAMIS
O. F. DEVEREUX
*Department of Metallurgy,*
*Institute of Materials Science,*
*University of Connecticut,*
*Storrs, Connecticut, USA*

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## *Some observations of glass-ceramic microstructures by cathodoluminescence*

The effects of variations of thermal treatment on the microstructures of glass-ceramics prepared by the controlled crystallization of glasses are of interest and, therefore, rapid and convenient methods of characterizing the microstructures are required. The scanning electron microscope (SEM) has become an important tool in the investigation of the surfaces of materials; there are three basic modes in which the SEM can be operated to observe the interactions of an electron beam with the surface of a specimen. These are the electron emission, the X-ray emission and the photon emission from the specimen surface [1].

In this letter we describe some results obtained from observing the cathodoluminescence (CL) or photon emission from a glass-ceramic. The system used to detect the CL consisted of an EMI Type 6255B photomultiplier tube and a 1 in. diameter lens of focal length equal to 1 in. to give a collection angle in excess of 0.75 steradians; a Cambridge Scientific Instruments Ltd, Mk IIA Stereoscan was used.

Two glasses were prepared from compositions shown in Table I; glass A was heat-treated for 2 h at 800°C [2] and glass B was extruded at 825°C. Both glass-ceramics contained needle-shaped crystals of lithium disilicate as their major crystalline phase and a crystalline form of silica as a minor crystalline phase, together with a residual glass phase. Specimens approximately 3 mm thick of each material were polished to 1 μm and coated with a layer approximately 10 nm thick of gold/palladium to prevent charging in the SEM. The polished surfaces were examined in the SEM by the secondary emission mode and the CL mode. The accelerating potential was 30 kV, the beam current approximately 150 μA and the specimen was positioned normal to the incident electron beam.

TABLE I Glass compositions in mol %.

| | $SiO_2$ | $Li_2O$ | $K_2O$ | $Al_2O_3$ | $P_2O_5$ | $B_2O_3$ |
|---|---|---|---|---|---|---|
| Glass A | 61.0 | 30.5 | 1.5 | 1.0 | 1.0 | 5.0 |
| Glass B | 67.5 | 24.0 | 1.5 | 1.0 | 1.0 | 5.0 |

Figs. 1 and 2 show a comparison of the same area of glass A observed in the two modes; Fig. 3 shows a sample prepared in the same fashion but etched for 30 sec in a 3% HF solution and observed in the secondary/reflected mode. There is obviously a marked contrast mechanism in the CL mode which enables the lithium disilicate crystalline phase to be easily distinguished from the glassy matrix; the CL micrograph has a speckled appearance because of noise associated with the photomultiplier.

Figs. 4 and 5 show a similar comparison in glass B. A weak contrast mechanism can be observed in the secondary/reflected emission micrograph which is caused by the back-scattered electrons [3]; the darker crystalline regions in Fig. 4 show little electron emission but a strong photon emission in Fig. 5. Prior etching of the glass-ceramic, in the manner described above, enabled the micrograph shown in Fig. 6 to be obtained by the secondary/reflected mode.

CL has been observed in quartz [4] but in the present studies no CL was observed from the silica crystalline phase; one of the emission bands in quartz [5] is positioned outside the range of the photomultiplier tube used and the strong emission from the disilicate phase together with the small volume fraction of the silica crystals makes detection of the CL of the latter difficult.

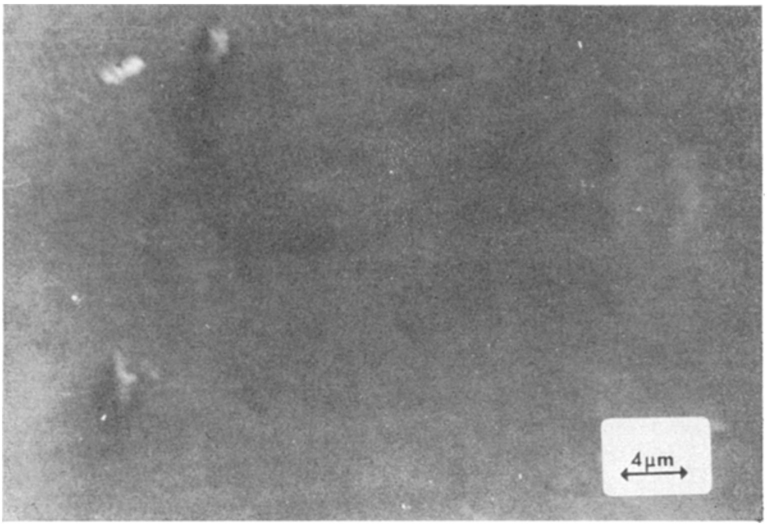

*Figure 1* Secondary/reflected emission from a polished sample of glass A.

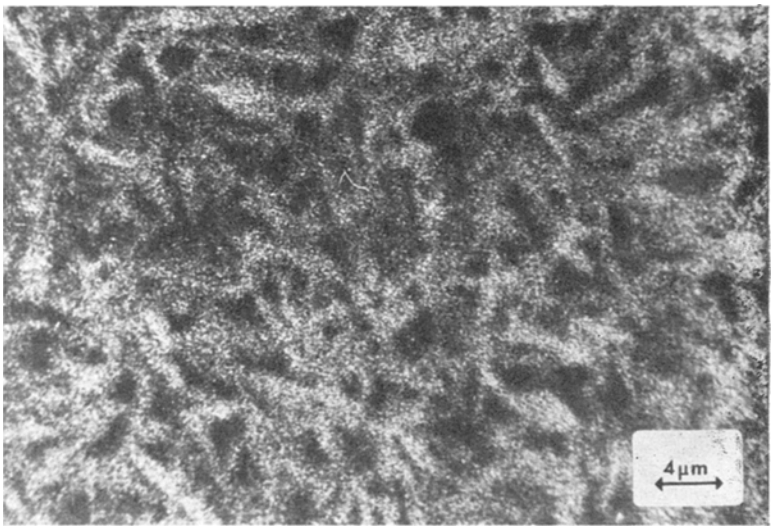

*Figure 2* CL from the same area as in Fig. 1.

*Figure 3* Secondary/reflected emission from etched sample of glass A; A is the silica crystalline phase.

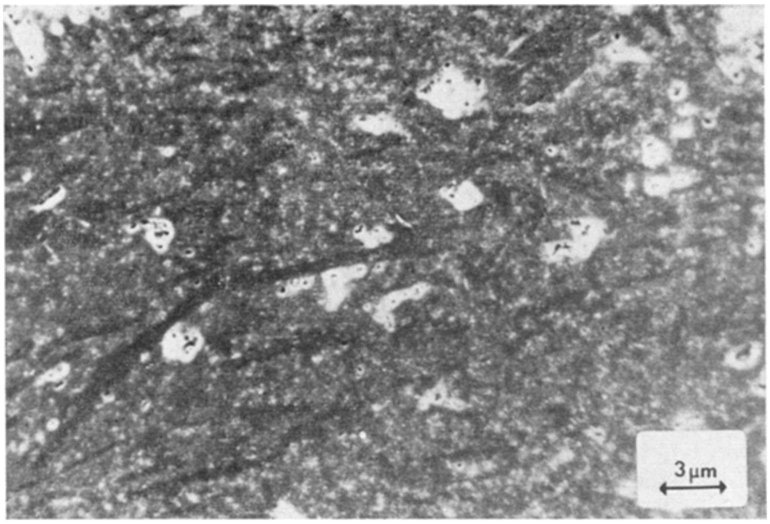

*Figure 4* Secondary/reflected emission from a polished sample of glass B.

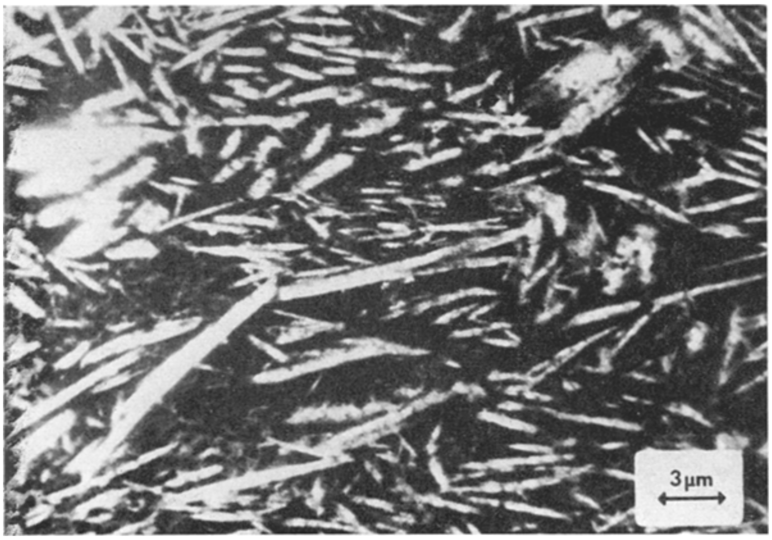

*Figure 5* CL from the same area as in Fig. 4.

*Figure 6* Secondary/reflected emission from an etched sample of glass B: A is the silica crystalline phase and B is the disilicate phase.

It is clear from the present work that observation of CL can provide an additional and valuable technique for the investigation of glass-ceramic microstructures. In this study the CL observed was confined to lithium disilicate crystals. However, this had the advantage of enabling these crystals to be readily distinguished from the crystalline silica phase. With an improved system for detection and analysis of the radiation emitted, it may be possible to extend the technique to selectively reveal the morphology of the silica crystals and of other crystal types in different glass-ceramic compositions. As the micrographs indicate, CL enables micrographs showing good contrast to be obtained without the necessity for etching of the glass-ceramic surface. The latter could cause modifications of the microstructure and clearly this is undesirable.

We conclude that the technique of using CL gives additional valuable information in the investigation of polished glass-ceramic surfaces and also has a considerable potential for characterizing the fracture surfaces of multiphase materials based on elements of low atomic number.

## Acknowledgements

The authors would like to thank Mr G. Smith for invaluable assistance with the electron microscope. Gratitude is also expressed to the SRC for financial support.

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*Received 26 November*
*and accepted 27 November 1973*

D. I. H. ATKINSON
P. W. MCMILLAN
*Department of Physics,*
*University of Warwick,*
*Coventry, Warwickshire, UK*

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## *Possible use of glass-ceramics containing a crystalline ferrimagnetic phase as memory material*

Several publications have now appeared in the technical press describing the magnetic properties of glass-ceramics containing crystalline ferrites [1-10]. A number of ferrite and glass forming systems have been investigated and in several of these publications the magnetic properties of lithium ferrite glass-ceramic have been studied. Lithium ferrite, $LiFe_5O_8$, is a magnetic material of commercial interest and is employed as a basis for square *B-H* loop material which can be used for the manufacture of memory element toroids and which displays stable magnetic properties over a wide range of operating temperature. A necessary condition for a material to be considered for application as memory material is that the material should display a high degree of loop squareness. This is particularly important when the elements are assembled into a 3D organization where the $\delta$ noise arising from the $\frac{1}{2}$ select currents passing through cores on the same $x$ and $y$ lines as the core being interrogated must be minimized. The purpose of this note is to point out that glass-ceramics containing crystalline ferrite are not, in general, suitable as square loop material.

A glass-ceramic containing crystalline ferrite can be considered to act as an assembly of ferrite particles held together by a glass binder. The degree of squareness attainable with a glass-ceramic is limited to a value below that of the pure ferrite due to the presence of the non-magnetic glass binder which causes the glass-ceramic to be subject to an internal demagnetizing field. This field has the effect of altering the shape of the loop and reducing the squareness. The internal demagnetizing field, $H_{\mathrm{int}}$, is given by

$$H_{\mathrm{int}} = -NM$$

where $N$ is a constant depending on the volumetric fraction of glass in the glass-ceramic and $M$ is the magnetization. It is possible to make an estimate of the size of the demagnetizing field from data on $Na_{\frac{1}{2}}Zn_{\frac{1}{2}}Fe_2O_4$ published by Smit and Wijn [11]. Assuming that in the absence of internal demagnetization the material displays a