indicating that the behaviour at very high temperatures could be even more complex.

Mizuno and Noguchi [3] also reported that the YAP phase was unstable when heated at temperatures below 1600°C for periods of the order of 20 h, and assumed its stable region to be above this temperature [9]. They did not specify whether powder or bulk material was used. The YAP phase is now usually assumed to be a congruently melting compound mainly because it has proved possible to produce single crystals by Czochralski growth. While the twinning and cracking found to occur in these bulk crystals may be related to this thermal degradation detected in powders, it seems more reasonable to attribute these effects to the observed anisotropic thermal expansion behaviour of this phase [10]. Bulk crystal chips of the present material showed no evidence of decomposition when subjected to similar heat treatments as the powdered samples, indicating that the reaction kinetics are strongly dependent on the surface to volume ratio of the sample. Thus, the reaction clearly proceeds much more slowly in the bulk than in the powdered material with the result that it has no significant effect during the time usually involved in the growth of single crystals by the Czochralski method. However, crystals deliberately held for prolonged periods at high temperature after growth tend to develop optical scattering centres and it is possible that the generation of these defects is related to the decomposition observed in the present material. This is one of the aspects of this reaction which is at present under investigation.

Acknowledgements

The authors wish to thank Messrs B. Lent and J. G. Plant for their valuable assistance in the preparation of the samples, and Professor R. E. Smallman for the provision of some of the laboratory facilities. This paper is published by permission of the Copyright Controller, HMSO.

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10. B. COCKAYNE, B. LENT, A. W. VERE, J. S. ABELL and I. R. HARRIS, to be published.

Received 17 March
and accepted 26 April 1972

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Neodymium-doped glass-ceramic laser material

Since the time Maiman [1] demonstrated the first successful ruby laser, numerous transition metal and rare-earth-doped materials have been used as solid-state lasers. All these materials can be classified as either single crystals or as glasses. The reason for this, of course, is that most transparent inorganic materials fall into these two classifications.

This letter describes the first reported lasing of a transparent glass-ceramic host. The significance of this is the lasing of a two-phase material consisting of an anisotropic crystalline phase as well as a second isotropic glassy phase. The glass-ceramic host has some desirable physical properties for laser materials, such as the very low thermal expansion.

The glass from which the glass-ceramic was prepared has the following compostion in mol %: 73.14% SiO_2, 13.73% Al_2O_3, 8.69% Li_2O, 1.75% BaO, 1.51% TiO_2, 1.08% ZrO_2, and 0.10% Nd_2O_3. The glass was melted at 1600°C

TABLE I X-ray data of the high quartz solid-solution phase in the Nd^{3+} doped glass-ceramic

$a_0 = 5.180$			$c_0 = 5.450$		
dÅ	I/I_1	hkl	dÅ	I/I_1	hkl
4.473*	13	100	1.73	2	202
3.460	100	101	1.69	1	210
2.603*	5	110	1.616*	9	211
2.324*	1	102	1.494*	2	300
2.238*	4	200	1.438*	5	212
2.071*	4	201	1.410*	5	203
1.875*	15	112			

*Used to calculate a_0 and c_0.

and annealed. A portion of the annealed glass was heat-treated to produce the glass-ceramic.

An X-ray diffraction pattern was obtained on the heat-treated glass-ceramic in order to identify the phases present and to estimate the extent of crystallization. The percentage of glass was estimated to be 25% by comparing the intensity of the glassy hump for the glass-ceramic to the intensity of the glassy hump for the original glass [2]. The two major peaks for cubic ZrO_2 were also detected, but this phase does not represent more than about 1% of the material since this was the original amount of ZrO_2 present. The only other phase detected was the high-quartz solid-solution phase which is commonly observed in Li_2O-Al_2O_3-SiO_2 glass-ceramics [3, 4]. This phase represents about 75% of the glass-ceramic. The d-spacings, relative intensities and Miller indices, along with the unit cell dimensions for this phase are given in Table I.

Fig. 1 shows an electron micrograph of the glass-ceramic after being etched for 4 min in a $\frac{1}{2}$% HF – 2% HCl solution in ethanol. It appears from the micrograph that most of the crystals in the glass-ceramic are about 100 to 300Å in diameter.

TABLE II Thermal expansion and laser threshold measurement for Nd^{3+} doped glass and glass-ceramic rods

	$\alpha(\times 10^{-7}/°C)$ (0 to 300°C)	Laser threshold (joules)
Unheat-treated glass	39.5	
Rod No. 1		48
Rod No. 2		37
Glass-ceramic		
Rod No. 1	− 4.1	99
Rod No. 2	− 4.9	91
Rod No. 3	− 5.2	75
Rod No. 4	− 5.5	91

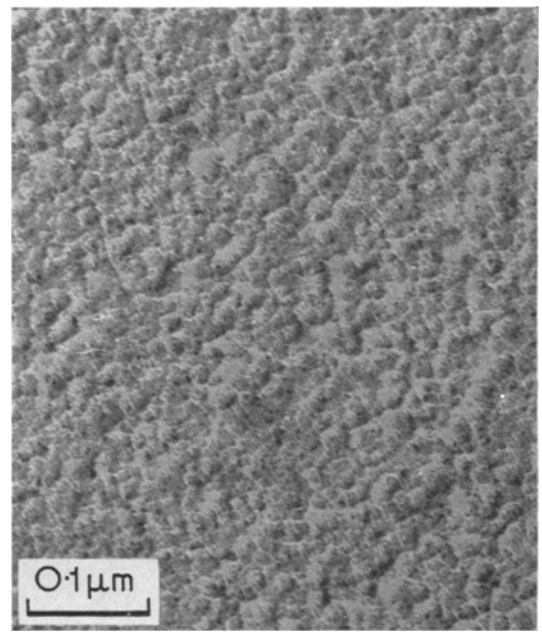

Figure 1 Electron micrograph showing the microstructure of the Nd^{3+} doped glass-ceramic (etched for 4 min in a $\frac{1}{2}$% HF-2% HCl solution in ethanol).

Four rods, $\frac{1}{4}$ in. in diameter by 3 in. long, were cut from the heat-treated glass-ceramic block. The coefficients of thermal expansion were measured for each of the rods and they were found to be about $-5 \times 10^{-7}/°C$ as compared to $39.5 \times 10^{-}/°C$ for the original glass. The measured values for each of the rods are given in Table II.

The ends of the four glass-ceramic rods used for the thermal expansion measurements, as well as those of the two similar rods cut from the original glass, were finished to a flatness of about one-tenth the wavelength of the visible light and they were made parallel to within 10 sec of arc for the laser measurements. Laser measurements were carried out in a silver elliptical cavity with external dielectric mirrors. The laser rods were pumped with an ILC-4L3 xenon flashlamp connected to a 200 μfd capacitor bank. A 200 μh inductance connected in series with the capacitor, gave rise to a pumping flash of about 300 μsec. To determine the lasing threshold for each rod, the output of the laser was measured by an S-1 vacuum photodiode through a 1060 nm narrow band pass filter. The threshold for lasing was taken as the pump energy at which spiking, a typical characteristic of pulsed solid-state lasers,

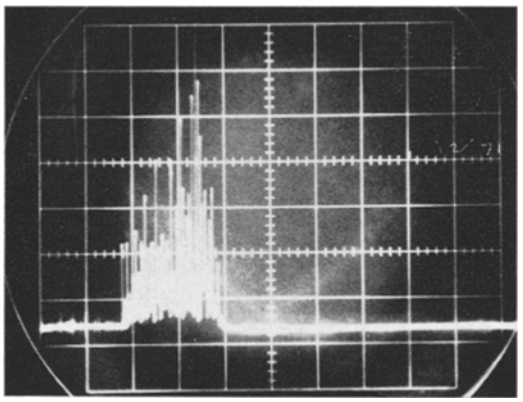

Figure 2 Oscilloscope trace showing the laser output of the Nd^{3+} doped glass-ceramic laser rod No. 3 pumped at 169 joules (scan = 100 μsec/div).

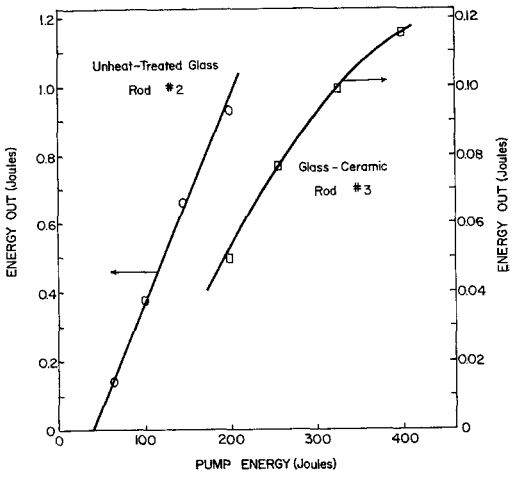

Figure 3 Plot of laser energy out versus pump energy for glass and glass-ceramic laser rods.

was detected. The oscillogram in Fig. 2 shows the spiking characteristics in the output of the glass-ceramic laser rod No. 3 pumped at 169 joules (about twice the threshold). With an output mirror reflectivity of 74.5%, the threshold for the glass-ceramic laser rods was found to be about two to three times as high as that of the glass laser rods; that is, about 90 joules for the glass-ceramic as compared to about 40 joules for the glass. The measured thresholds for the glass-ceramic and glass laser rods are given in Table II. The lasing efficiencies of the glass-ceramic rod No. 3 and the glass rod No. 2 were measured with an output mirror reflectivity of 83.5%. Output energy measurements were made with a TRG model 101 thermopile. Fig. 3 shows a plot of the energy output versus the pump energy for these two laser rods. As can be seen, the lasing efficiency of the glass-ceramic laser rod is lower by at least an order of magnitude relative to that of the glass. This is a considerable decrease in the efficiency considering the fact that the thresholds for these two rods differed by about a factor of 2.

Subsequent publications will treat the spectroscopic properties of the active ions in this host and the influence of the microstructure on the properties of this laser host.

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Received 4 April
and accepted 18 April 1972

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Surface effects before and after the aragonite-type to calcite-like transformation in potassium nitrate in relation to mechanism

In KNO_3 the aragonite structure II is known to change above 128°C to a calcite-like structure I with anions essentially coplanar but orientations disordered within the plane [1]. In the structure of II, symmetry *Pmcn*, the arrangement of cations is approximately as in hexagonal close-packing: the anions are in distorted simple hexagonal array. The arrangement of the ions in I (S.G. $R\bar{3}m$) is a rhombohedral distortion of that in NaCl [2], related to cubic close-packing.

The transformation II → I has been considered to produce no surface effects, except that recently Asadov and Nasirov [3] briefly reported traces parallel to the interface. Cahn [4] has mentioned the possible relevance of twinning of the