JOURNAL OF MATERIALS SCIENCE 5 (1970) 837-838

Further Developments in Oxide Crystal Growth using Gas Lasers

B. COCKAYNE, D. B. GASSON
Royal Radar Establishment, Malvern, Worcs, UK

N. FORBES
Ferranti Ltd, Silverknowes, Edinburgh, UK

It is shown that a DC energised CO_2–N_2–He laser system leads to a lower loss of material by evaporation than a comparable AC system when applied to oxide crystal growth. The reasons for this behaviour are discussed and a method for minimising evaporation using an AC system is described.

1. Introduction

In an earlier publication, two of the present authors [1] demonstrated the use of AC energised CO_2–N_2–He gas lasers, operating at 10.6 μm, as a heat source in the growth of high melting point oxide single crystals by a floating zone recrystallisation method. One of the difficulties encountered was the evaporation of material from the molten zone. It is the purpose of the present publication to report comparative measurements of evaporation losses using AC and DC energised lasers and to comment upon the differences which exist.

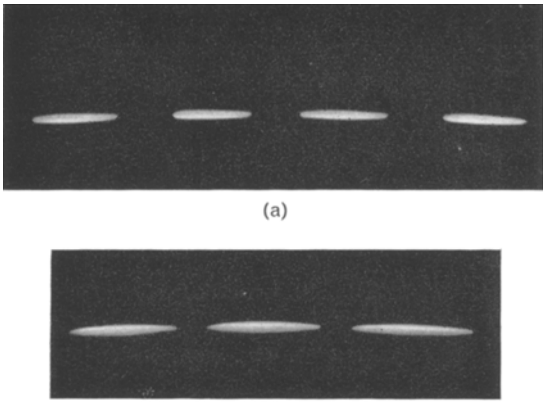
2. Experimental

The lasers used were both manufactured by Ferranti Ltd and were of the type in which the output power could be varied within the range 50 to 500 W. The conditions used to investigate evaporation losses were similar to those used in crystal growth. Sintered oxide charge rods, 6 mm in diameter and prepared as for crystal growth [1], were placed at the focus of the laser beam in order to establish a molten cap on the charge rod. The diameter of the laser beam incident upon the specimen was 0.15 cm in each case and focusing was achieved using the long focal length optics already described [1]. The specimen was rotated through the laser beam and the top was maintained in the molten state for periods of up to 5 min which corresponds to the time interval in which the material would be molten during floating zone recrystallisation at normal crystal growth rates (typically 2 to 3 cm h^{-1}). Evaporation losses were recorded by weighing the charge rods on a standard Mettler microbalance to an accuracy of 0.001 g before and after insertion in the laser beam.

3. Results and Discussion

The behaviour of two high melting point oxide materials was investigated. These were yttria (Y_2O_3; M pt 2450° C) which is only slightly volatile during crystal growth and neodymia (Nd_2O_3; M pt 2250° C) which is sufficiently volatile to inhibit single crystal growth, yielding polycrystalline material instead. The beam power of the AC and DC lasers was maintained at a constant output of 300 W under optimum tuning conditions for the comparative measurements and was standardised in each case using the same Ferranti total radiation calorimeter in conjunction with a chart recorder.

The results, given in table I, show that the evaporation rate is considerably reduced by the use of a DC energised laser instead of an AC system. This is consistent with the lower peak powers generated in a DC laser. In a DC laser the mean power is equivalent to the peak power so that 300 W in the primary beam represents a power density of 16.8 kW cm^{-2} when incident upon the specimen at a focus diameter of 0.15 cm. In an AC laser the applied voltage drops below the threshold voltage required to energise the lasing discharge for a fraction of each half cycle, giving rise to a "dead" time and a corresponding increase in power in the period when the laser is activated for any given power out-



(a)

(b)

Figure 1 Streak photographs of a focused AC laser at mean power outputs of (a) 300 W, (b) 500 W.

put. An estimate of the dead time can be obtained by quickly passing a perspex sheet through the focal point of the converging laser beam. This marks the perspex in a manner which can be reproduced as a streak photograph, shown in fig. 1, thereby allowing the dead time and activated time to be calculated for any half cycle. (One half cycle = 10 msec.) Fig. 1 shows streak photographs corresponding to measured power outputs of 300 and 500 W giving dead times of 3.3 and 2.3 msec respectively. In order to maintain 300 W output power, the mean power during the remaining 6.7 msec must be 450 W which, assuming a sinusoidal power distribution, corresponds to a peak power of 625 W for the AC system. This is equivalent to a power density of 35 kW cm^{-2} at a focus diameter of 0.15 cm, which is substantially higher than that experienced in the DC system. At 500 W output power, a similar calculation shows that the peak power at the focus is 50 kW cm^{-2}. It should, however, be noted that peak power is not increased by the same factor as the output power, due to the decrease in dead time as the output power is increased. This decrease in dead time arises from the need to increase the gas flow rate through the laser cavity as well as raising the voltage, in order to increase the laser beam power under optimised operating conditions. The rise in gas pressure within the cavity causes laser action to occur at a lower voltage, i.e. earlier in any half-cycle. The calculated peak power values are probably too high by a factor of 5 to 10 % as the streak photographs suggest the existence of a slight plateau rather than a maximum in the power distribution through each half cycle.

The mean power values required to obtain a molten zone during crystal growth can be reduced by raising the specimen temperature. This reduces the peak power incident upon the specimen when an AC laser system is employed and leads to a lower evaporation rate. For instance, the evaporation rate for Y_2O_3 is reduced to 0.024 g min^{-1} by raising the ambient temperature to approximately 1750°C and using a mean power output of 200 W under comparable experimental conditions to those used for obtaining the results given in table I.

TABLE I

Materials	Evaporation rate AC laser	(g min^{-1}) DC laser
Y_2O_3	0.076	0.001
Nd_2O_3	0.272	0.041

Mean beam power = 300 W in all cases.

4. Conclusions

It may be concluded that evaporation losses during oxide crystal growth using CO_2–N_2–He gas lasers are minimised by using a DC energised rather than an AC energised system. However, evaporation losses using an AC system can be reduced by raising the ambient temperature of the crystal. One additional point which arises from the present work is the usefulness of the AC system for machining oxide and related ceramic materials which absorb 10.6 μm radiation. In this case the higher evaporation loss is an advantage.

Acknowledgement

This paper is published by permission of The Copyright Controller, HMSO.

References

1. D. B. GASSON and B. COCKAYNE, *J. Mater. Sci.* **5** (1970) 100.

Received 22 June and accepted 18 July 1970